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University of Alberta,

Edmonton, Alberta,

1924 ----- 1925

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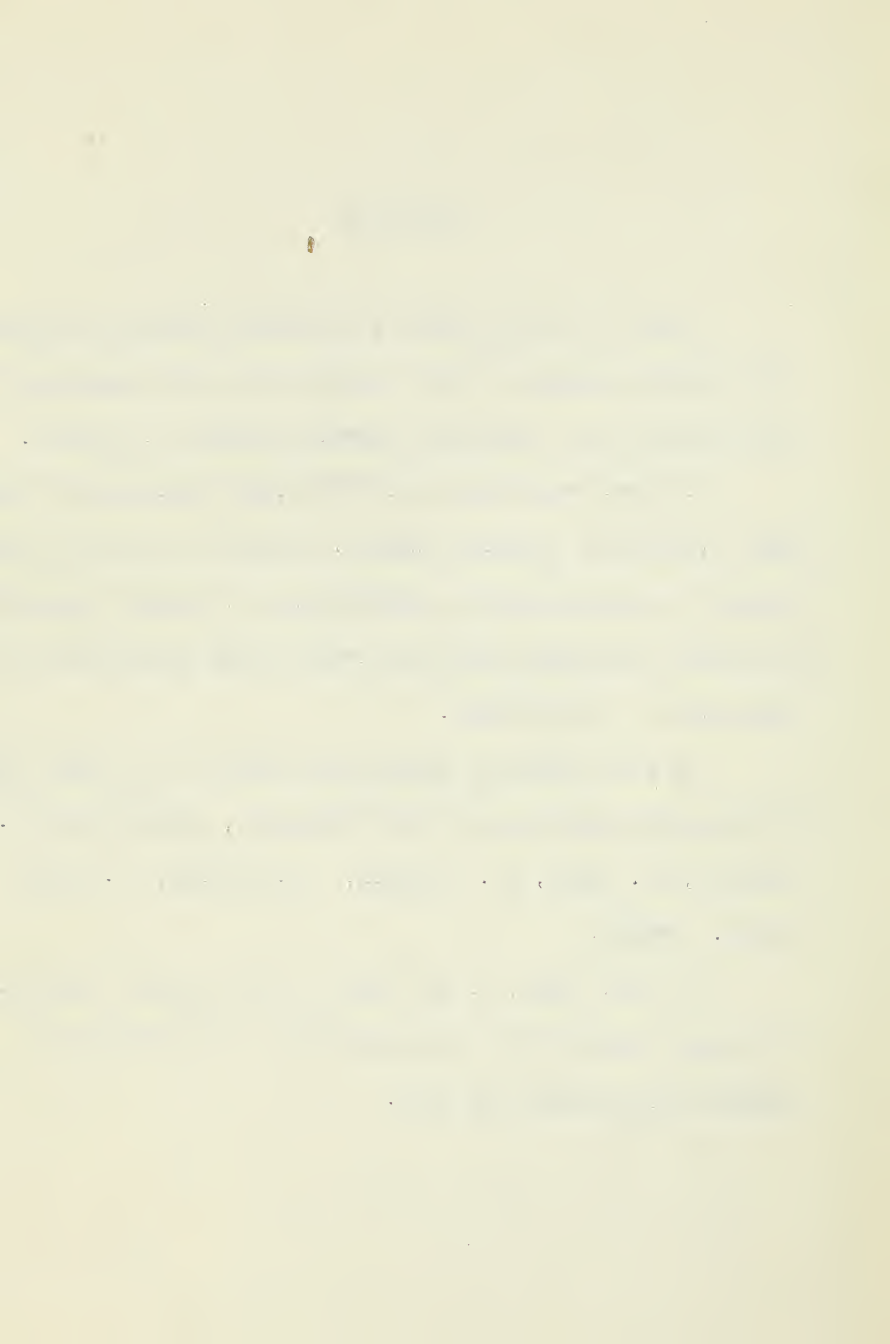
PREFACE

Much of the work dealt with in this thesis has arisen from routine analyses of coal carried out in the Laboratory of the Scientific and Industrial Research Council of Alberta.

The work was done under the direct supervision of Mr. Edgar Stansfield, Research Engineer in Fuel, to whom the writer desires to acknowledge his indebtedness for helpful suggestion and kindly criticism which have been of much assistance in the preparation of this thesis.

He also wishes to thank many members of the Staff of the Research Council and of the University, particularly Mr. Hollies, Dr. Clark, Dr. Lehmann, Mr. Pitcher, Dr. Boyle and Mr. Jewitt.

To Mr. Blair, of the Staff of the Research Council, he is much indebted for the preparation of the graphs and drawings illustrating the work.



SOME PROBLEMS IN COAL ANALYSIS

GENERAL INTRODUCTION

It soon becomes apparent in analysing coal that an exceedingly unstable substance is being dealt with. W. A. Noyes ⁽¹⁾ says, "It cannot be emphasized too strongly that in coal we have an extremely complex mixture of organic compounds, many of which are easily changed by oxidation and otherwise by exposure to air." Even sealing coal in an air-tight container does not stop the processes of change; in short, coal cannot be preserved in the strict sense of that word. Evidence of this is not lacking, for it has been observed that some coals when sealed in vessels will generate a pressure within the vessel, and others will create a vacuum. The former condition, no doubt, points to a breaking down of the coal substance into simpler compounds, while the latter indicates absorption of the gases over the coal. J. W. Lewis ⁽²⁾

⁽¹⁾ Journal Ind. Eng. Chem., June 1913, Vol.5, p.517.

⁽²⁾ "A further study of Alberta coal." U. of A., J. W. Lewis, Thesis for M.Sc. degree.

had noted that the moisture content of ground coal, kept in glass bottles with ground-in stoppers, tends to increase with time; these tests have been elaborated on, and the findings confirm Lewis' observations.

It would appear that once coal has been removed from its natural surrounding, changes begin to take place immediately.

It is evident that in the analysis of such a changeable substance as coal, the results for a particular coal will depend on: (1) the nature of that coal, (2) the time which has elapsed since mining, and (3) the conditions under which the coal has been stored. A coal fresh from the mine will give different results from a coal, taken from the same mine and seam, which has been exposed to weathering for some time. Nor do these conditions alone influence the results obtained on analysis of coal, but also such conditions as humidity and temperature. These things being so, the necessity of standardized methods of analysis becomes evident. Even the apparently simple matter, the determination of the moisture in coal, depends on the conditions under which the test is carried out. Illingworth ⁽¹⁾ has said: "In any specification which relates to the purchase or supply of coal, it is essential to detail the method to be adopted in determining the moisture." Coals, then, which are to be compared and classified must be analysed by the same methods, if the results are

(1) "The Analysis of Coal and Its By-Products," S. R. Illingworth, page 19.

to be comparable.

There are few coal fields in the world where, within so narrow confines, are to be found the variety of types of coal found in Alberta. The coals range from semi-anthracite through the bituminous, sub-bituminous, to the black and brown lignite types. Almost the whole series, then, of common types of coal is represented in the Province, except cannel and, perhaps, anthracite. Claims have been made from time to time that anthracite has been found, but analyses do not support these claims.

In the work which is dealt with later, typical coals from the classes mentioned were used; and where coal is mentioned it refers to coal mined in the Province of Alberta, unless otherwise stated.

In the laboratory of the Scientific and Industrial Research Council of Alberta, where this work was carried out, it was essential in undertaking an extensive analysis of the coals of the Province that recognized standard methods of analysis should be employed if the work was to have any significance outside the Province. For this reason methods substantially those laid down by the A.S.T.M. were adopted. From time to time, however, departures in the direction of more accurate determinations have been made, or changes in the method that would bring out inherent properties of the coal without altering the purpose of the original method were introduced.

As the title may suggest, this thesis will be concerned with several topics, more or less closely related to each other, but all bearing directly on the general subject of "Coal Analysis." Groups of problems will be taken up under the following headings:-

CHAPTER I.- MOISTURE IN COAL.

- (a) Air drying by standardized method.
- (b) Method of determination of retained moisture.
- (c) Distribution of moisture in relation to ash.
- (d) Water of oxidation.

CHAPTER II.- MINERAL MATTER IN COAL.

- (a) Inert constituents in coal.
- (b) Water of hydration.

CHAPTER I.

MOISTURE IN COAL

INTRODUCTION

Coal analysis is divided into two classes:-

- (1) Proximate analysis,
- (2) Ultimate analysis.

The calorific value of a coal, though not a part of proximate analysis, is usually carried out in connection with it. Proximate analysis consists of the determination of the moisture, ash, volatile matter, and fixed carbon (by difference). It is a convenient way of assessing the value of a coal, but gives little or no information as to what the coal really consists of. An ultimate analysis, on the other hand, is more precise; it determines the elements which go to make up the coal substance, with the exception of those elements which constitute the ash. The elements so determined are carbon, hydrogen, nitrogen, sulphur, and oxygen (by difference). While determining the amount of these elements present, ultimate analysis does not give any clue as to how such elements are combined in the coal substance. Both types of analysis will be used in considering the problems on hand.

Moisture in Relation to Alberta Coals.- Perhaps it will not be out of place, before dealing with the problem, to make two general statements regarding the relation of moisture to Alberta coals.

(1) The moisture content of the coals varies all the way from less than 1% to almost 40%.

(2) The moisture content of the coals decreases progressively going from east to west in the Province.

Figure 1 ⁽¹⁾, prepared from several hundred analyses, shows these two facts very clearly.

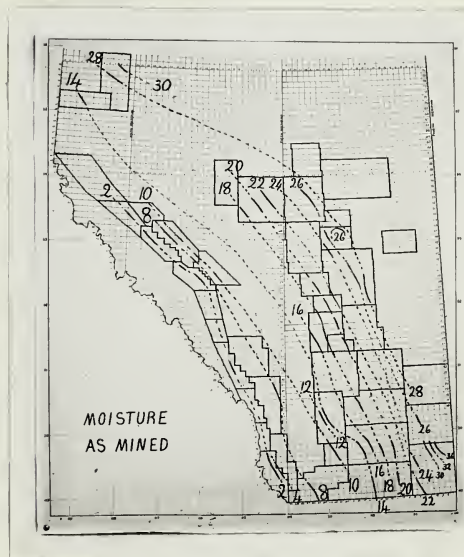


Fig.1.- Total Moisture Content of Alberta Coals as Mined, Percent.
Lines of equal moisture are plotted on a map of the Province of Alberta showing townships.

⁽¹⁾ Bulletin, C.I.M. & M., April, 1925, #156, p.412.

Physical Classification of Moisture in Coal

The first type of moisture, Surface Moisture, is always a variable quantity, its amount depending on the conditions in the mine. If the coal comes from a wet part of the mine, this loosely held surface moisture will be high, and vice versa. It may be looked upon as extraneous water, and it should be possible to remove it without disintegrating the coal -- in theory at least. It is not yet possible to say in the drying of a coal what amount of the total moisture is surface moisture alone. That it does exist, however, there can be no doubt.

The second type of moisture, "water of constitution", is the name applied to the combined and adsorbed moisture, and perhaps part of the adhering moisture in the coal. There are reasons for thinking that it is a fairly uniform quantity for coal over a small area. It varies, however, with the ash content, or perhaps more correctly, with the "pure" coal content. More will be said of this later. This type of water in high moisture coals cannot be removed without at least partial disintegration of the coal, but with coals of better quality the disintegration is less. On the gradual removal of water of constitution cracks develop in the coal body under ordinary laboratory and storage conditions, but a certain amount of moisture is retained by the slack from the disintegrated lumps, and is held quite tenaciously. In low moisture coal, water of

constitution is retained well, with little disintegration of the coal in drying under laboratory conditions.

The third type, "water of hydration," is the water retained by the mineral constituents of coal, such as clays, shales and gypsum. Its removal can only be effected at temperatures so high that volatile compounds of the coal also will be driven off; hence any attempt at quantitative estimation is vitiated. Indeed, none of the three types just mentioned can be separately determined quantitatively by direct analysis. Surface moisture and water of constitution can be determined together, their sum being called "total moisture ", though water of hydration is not included. A possible method for the estimation of surface moisture has been suggested, but time would not permit acting on the suggestion. If surface moisture could be determined, water of constitution could be found by difference, knowing the total moisture.

Practical Classification of Moisture in Coal

From the point of view of analysis, moisture in freshly mined coal falls under three heads:-

- (1) Moisture lost on air-drying,
- (2) Retained moisture.
- (3) Moisture in inert volatile matter.

The first type, as its name suggests, is the moisture given off

when the coal is air-dried. The second is the moisture still left in the coal after air-drying. These two combined are called the total moisture, and are equal to surface moisture plus water of constitution. The third type corresponds to water of hydration of the previous classification; it is always included in the volatile matter, though not a part of it. A fourth type is the moisture which appears to form in ground coal kept in glass bottles with ground-in stoppers. This, of course, is not a constituent of freshly mined coal; but it would appear that in certain types of coal, at least, its formation begins quite soon after the coal has been ground. The name, "water of oxidation" has been suggested. These four types of moisture will be treated in separate sections.

AIR-DRYING BY STANDARDIZED METHOD

Coal, in being prepared for analysis, is usually subjected to the treatment of "air-drying." In the case of low moisture coals, the treatment is optional, but in high moisture coals it is essential. It is practised for the following reasons, tersely stated by E. Stansfield ⁽¹⁾:-

- (1) To make it possible to grind the coal to powder.
- (2) To bring the coal sample to a condition in which it will not change in weight rapidly, whilst portions are

(¹) Trans. C.I.M. & M., #139, Nov. 1923, p.615.

being weighed out for analysis -- in other words, to bring it into equilibrium with the air in the laboratory.

(3) To determine the water retaining power of the coal."

The first two purposes may be attained by simply exposing the coal in open pans in the laboratory, until it ceases to lose appreciable weight. The loss of moisture by the exposed coal, then, depends on the humidity of the laboratory.-- high when the humidity is low, and low when the humidity is high -- so that the same coal dried under different conditions of humidity would lose different amounts of moisture. Figure 2 (next page) shows how the humidity does change from one season to another. The humidity shown was taken by means of a recording hygrometer.⁽¹⁾ This method of air-drying would satisfy the first two requirements, but not the third.

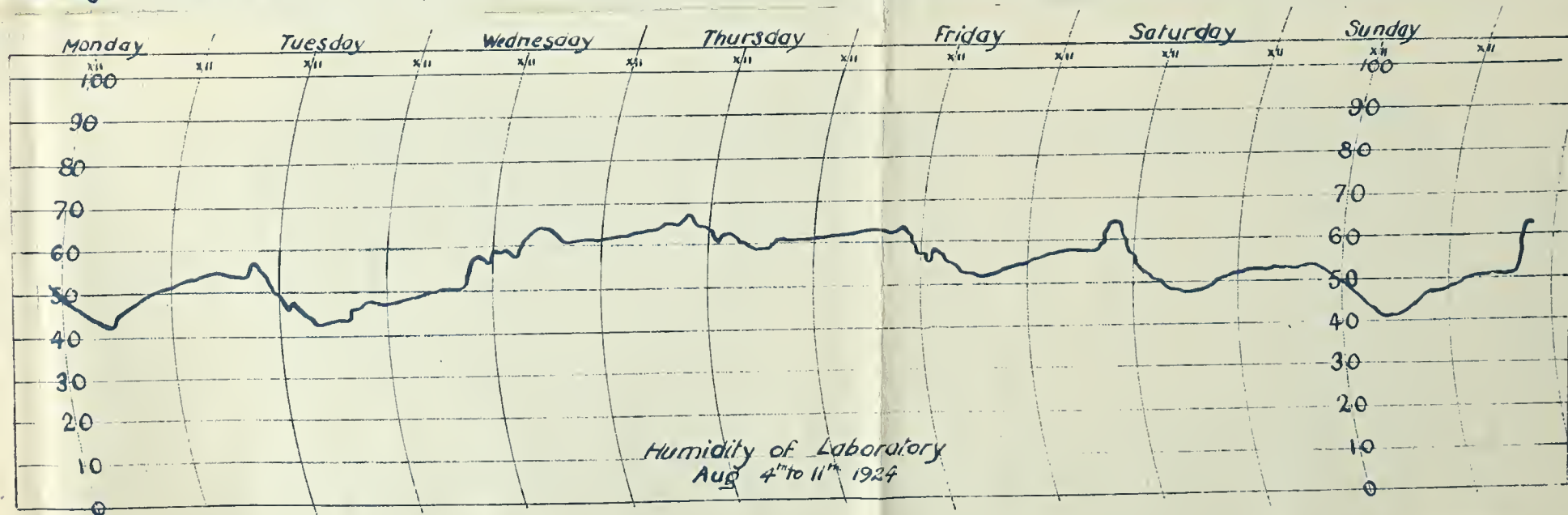
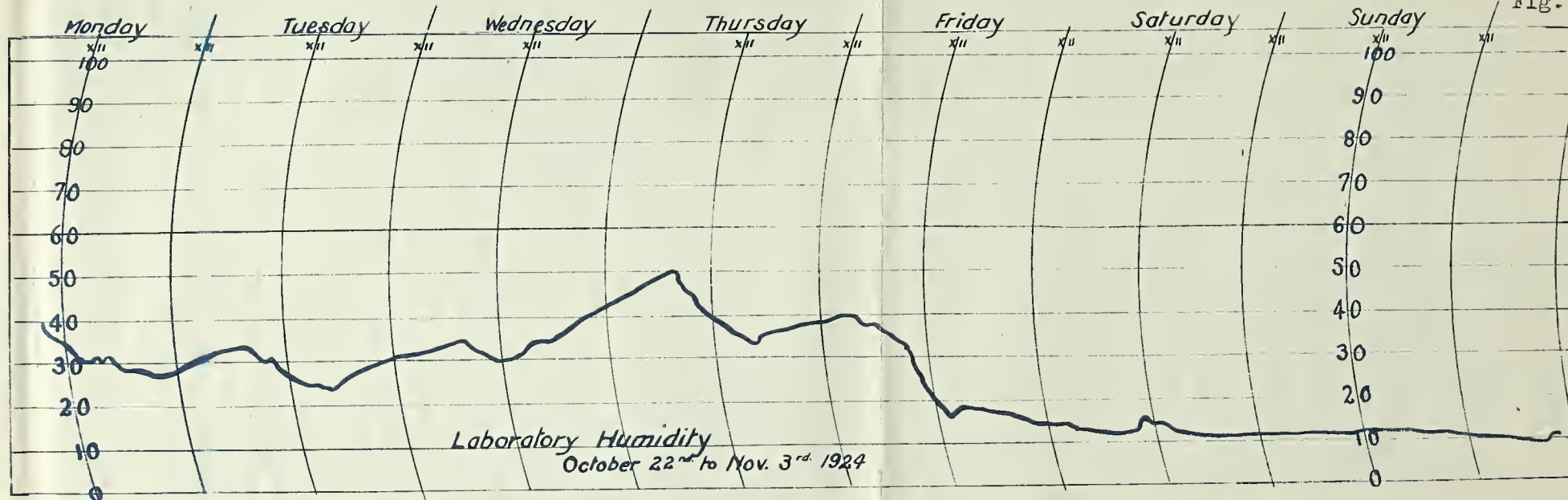
H i s t o r i c a l

So far as could be found out, the methods in general use only take into consideration the drying of the coal. The American Society for Testing Materials suggests exposing the coal in a special type of drying oven ⁽²⁾ maintained at 10 to 15°C. above room temperature till the loss in weight is not more than 0.1% per hour, but exposing in shallow pans in the laboratory is also permissible, and standard practice. Evidently only drying the

(1) Negretti & Zambra, London, England.

(2) A.S.T.M., 1924, p.982.

Fig. 2.



coal is thought of -- that is, the moisture lost, not the retentive power of the coal.

In the spring of 1907, at Mc Gill University, a survey of the coals of Canada, under the authority of the Dominion Government, was begun. E. Stansfield, who was in charge of the work of the chemical laboratory, devised an apparatus for the ⁽¹⁾ purpose of drying coal in a standardized way. It was pointed out that coals from the same mine and seam would vary in their moisture content according to climatic conditions, but there was a definite figure to which the percentage of moisture tended to approximate when coal is left exposed to the air but protected from rain. Weather conditions affect this percentage, increasing in wet weather and decreasing in dry. It was to overcome the variable conditions of humidity that the methods were decided upon.

Again, in 1923, when a chemical survey of the coals of the Province of Alberta was undertaken, an improved type of the same apparatus, Figure 3 (next page), was put in operation in the laboratory of the Scientific and Industrial Research Council of Alberta at the University of Alberta. ⁽²⁾ The improved apparatus has an air circulatory system, and some other changes for the more efficient working of the apparatus have been made by the designer since its installation. The apparatus as it now

⁽¹⁾ Investigation of the Coals of Canada , Vol.II., Part IX.,
J. B. Porter and others, p.130.

⁽²⁾ Fourth Annual Report, S. & I.R.C.A., 1923, pp.39-41.

Fig.3.



is, serves two purposes:-

- (1) It almost nullifies variable humidity conditions in the laboratory.
- (2) It dries coals in a standardized way, readily duplicated, so that the moisture retained by the air-dried coal would be characteristic of the particular kind of coal.

This apparatus, its structure and working, is described in full detail in the Bulletin of the C.I.M. & M.⁽¹⁾, and in the Fourth Annual Report of the Scientific and Industrial Research Council of Alberta (1923). It will be sufficient here to state that the apparatus is a closed system, consisting of two boxes connected by wide air pipes. (See Figure 3, preceding page.) The upper box has a rack for trays; the lower contains a fan which circulates the air through the system. The ascending air is moistened by a stream of calcium chloride solution of 1.30 specific gravity, or 700 grams calcium chloride, with two molecules of water of crystallization, per litre. This solution has a vapor pressure of approximately 60% that of water at laboratory temperature, or it will produce a relative humidity of 60%. The moistened air passes over the coal, and returns to the fan chamber. The designer's statement in the report just mentioned, regarding the solution, should be noted, viz.: the selection of 60% relative humidity was arbitrary. The coal sample is exposed for 48 hours

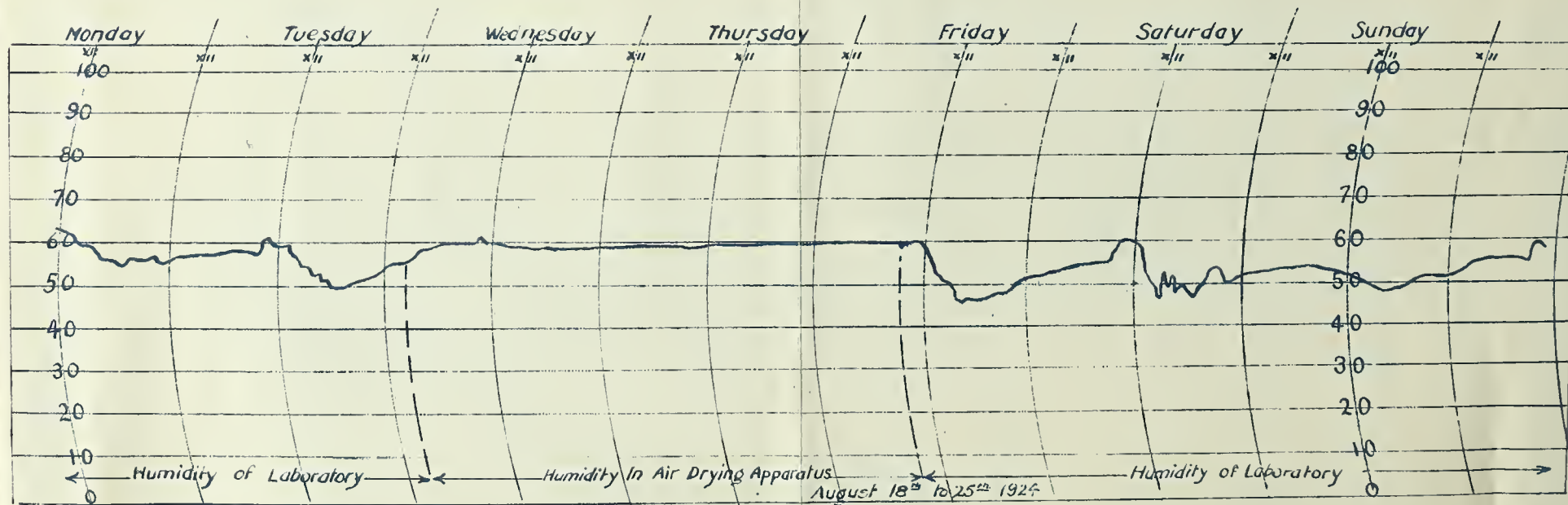
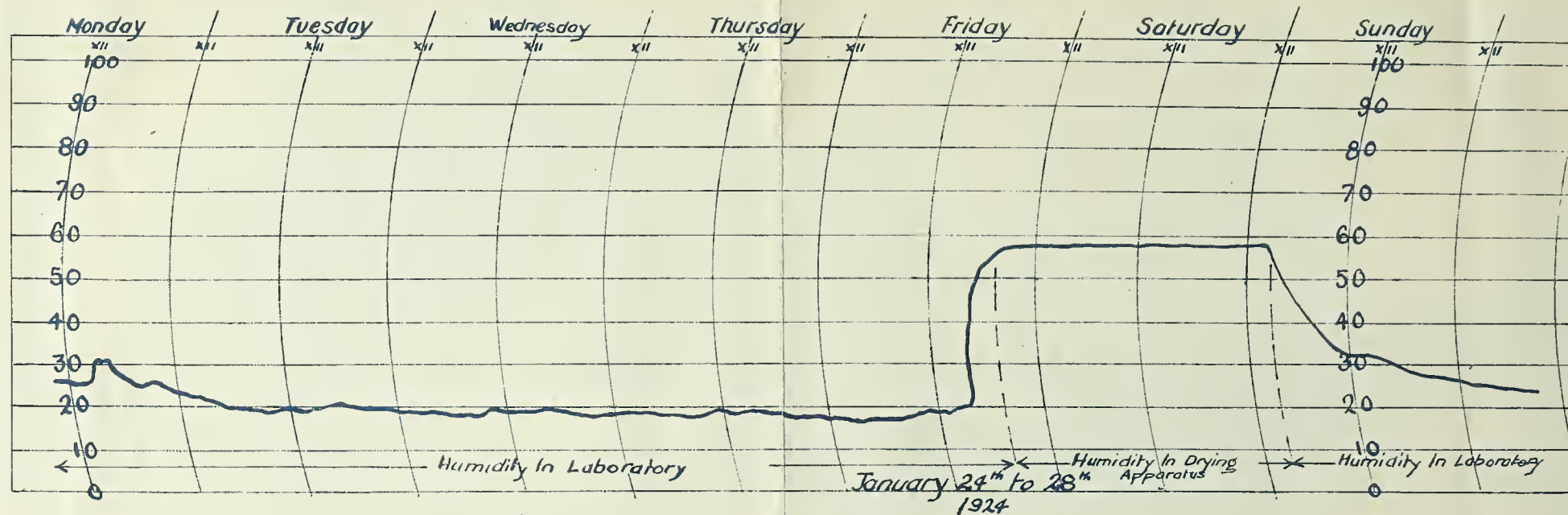
⁽¹⁾ Trans. C.I.M. & M., XXVI., #139, Nov. 1923, p.615.

in the apparatus, then weighed, the loss of weight being the loss of moisture on air-drying.⁽¹⁾ The reasons for 48 hours being chosen for the drying period are given in the report mentioned.

E x p e r i m e n t a l

Humidity in Air-Drying Apparatus.- The accompanying charts (Figure 4, next page) show the humidity inside the drying chamber under ordinary running conditions. All tests of the apparatus carried out by the writer have shown a steady condition of humidity, although not necessarily a relative humidity of exactly 60% in every case, as low as 50% being registered under somewhat adverse conditions. It has been noticed that during the cold months of the year, particularly December, January and February, the moisture retaining properties of the similar coals dried by this method are less than at other times of the year. This is largely due to the excessively low humidity of the laboratory caused in part by the relatively high temperature inside the laboratory as compared with the temperature outside the laboratory. Temperature, too, as well as humidity, in the laboratory has a slight effect on the satisfactory working of the apparatus.

⁽¹⁾ Under some circumstances, in air-drying by this method, coal may gain in weight, i.e., may take up moisture; but this has only happened when the samples were shipped in bags or leaky containers.



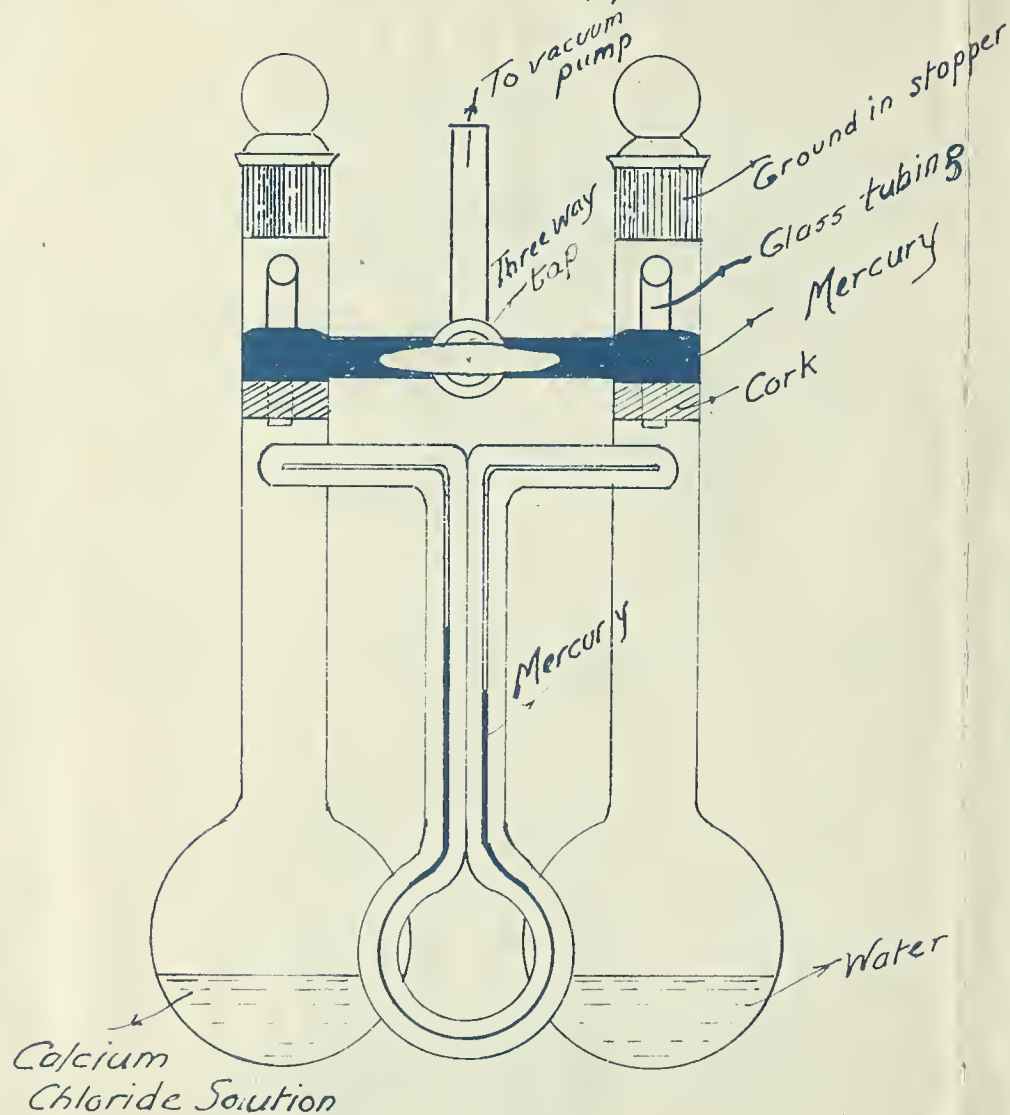
Vapor Pressure of Calcium Chloride Solution

Apparatus.- With a view to finding out what the variations in vapour pressure of the solution of calcium chloride were for different temperatures, experimental work was undertaken. The first attempts were unsuccessful, due to faults in the apparatus used.* The principle of the apparatus was sound, but stopper weaknesses prevented the vacuum, necessary in the determination, being maintained. It was designed to give directly the difference in vapour pressure in mm. of mercury between water and the solution of calcium chloride, with a specific gravity of that used in the "Constant Humidity Apparatus", viz., 1.30. Much time was spent, but no definite results were obtained.

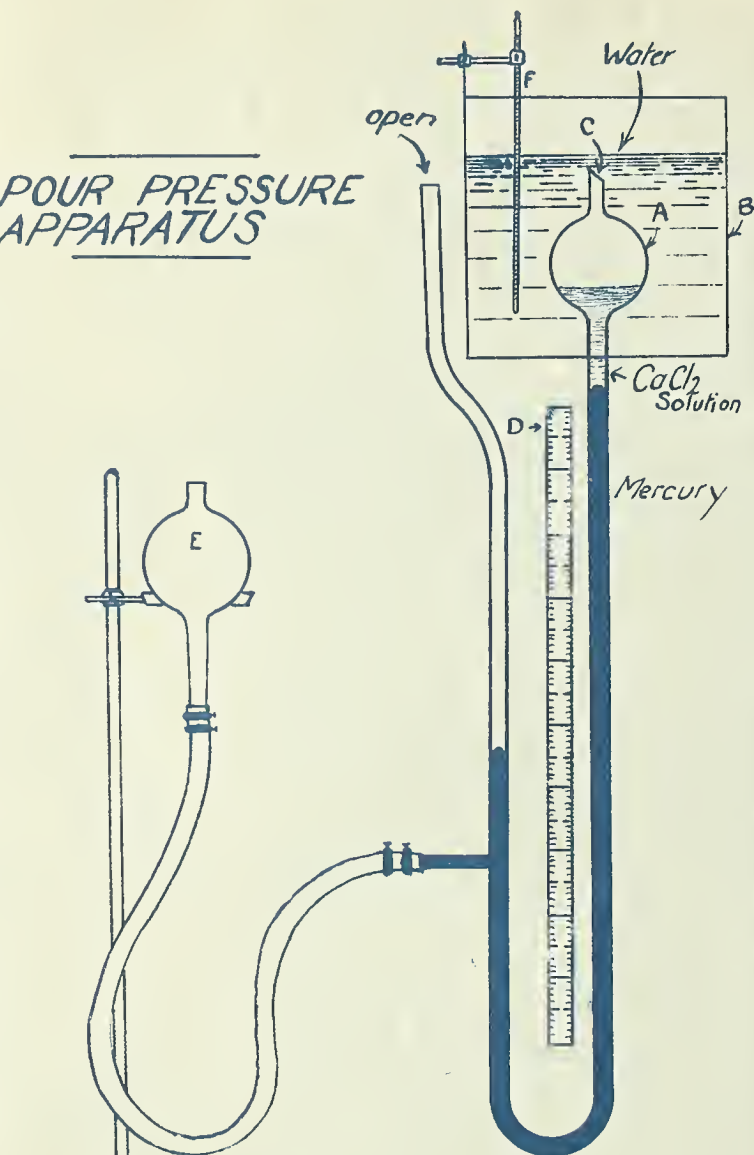
Through the kindness of Dean Boyle of the Department of Physics of the University of Alberta, a different type of apparatus was obtained. It was designed by Dr. Lang of the same department (Figure 5, next page). The apparatus consists of a vacuum bulb (A), blown on a U-column about 100 cm. long. The bulb is surrounded by a water bath (B), which can be heated through a range of temperatures. A scale (D) is placed between the arms of the U-tube, permitting readings being taken of the height of the mercury column in the right and left arms of the U-tube. A hydrostatic bulb (E) with rubber connection can be raised or lowered in order to keep the level of the mercury in the right-hand column at the point (F). The solution, in this

* Figure 4a, next page.

Vapour Pressure Apparatus.



VAPOUR PRESSURE APPARATUS



case Calcium Chloride of sp.gr. 1.30 at 20°C., the vapour pressure of which is to be determined, is introduced into the bulb (A), through the capillary at the top, a few cc. being ample. The bulb (E) is then raised till the solution, raised by the mercury column, goes into the capillary. The capillary is quickly sealed off with a blow-pipe, and the bulb (E) is lowered. Immediately the solution boils, and (A) is filled with its vapour. The apparatus is then ready for use. (A) is heated through a range of temperatures by the water bath (B). Readings are taken at definite temperatures, when steady for 15 minutes, of the heights of the mercury columns in the U-tube, the right-hand column always being kept at the point (F). The barometric pressure is also read for each reading. The vapour pressure of the solution is then the barometric pressure less the difference in height of the two mercury columns. A cathetometer which read to the nearest $1/20$ of a mm. was used in place of the less accurate scale shown in the figure.

Results.- The accompanying graph (Figure 6, next page) and Table I., from determinations made by the writer, show how the vapour pressure of the calcium chloride solution changes with a change in temperature. It will be seen from the table that a temperature between 18 and 22°C. should be maintained for the accurate operation of the apparatus, though the variation from 60% humidity is very small over temperatures from 14° to 32°C.

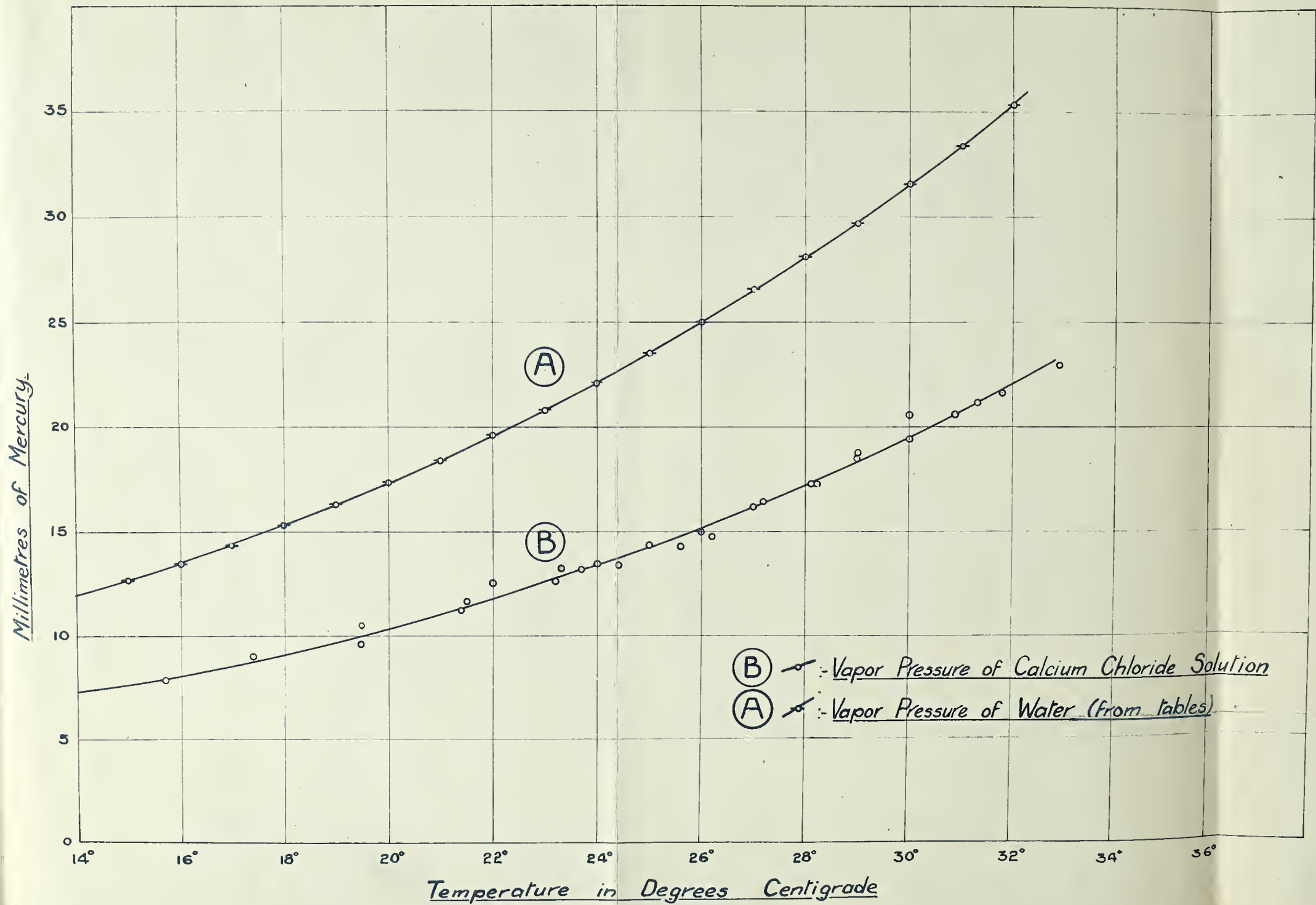


TABLE I.- VAPOR PRESSURE OF A SOLUTION OF CALCIUM CHLORIDE OF SPECIFIC GRAVITY 1.30 at 20°C.

Temp. °C.	Barometer mm.Hg	Cathetometer Readings		Diff'ce, mm.Hg.	Vapor Pressure of CaCl ₂ solution, mm.Hg.
		Closed Limb	Open Limb		
27.0	700.8	943.65	258.70	648.95	15.85
26.0	699.6	943.60	259.00	684.60	15.00

Tempera- ture, °C.	Vapor Pressure mm.Hg.	Tempera- ture, °C.	Vapor Pressure, mm.Hg.	Tempera- ture, °C.	Vapor Pressure, mm.Hg.
14.6	7.65	23.3	13.25	27.0	16.55
15.7	7.95	23.7	13.35	28.1	17.35
17.4	9.05	24.0	13.50	28.2	17.40
17.5	8.75	24.4	13.45	29.0	18.60
19.5	9.65	25.0	14.40	29.0	18.85
19.5	10.50	25.3	13.70	30.0	20.65
19.5	10.85	25.6	14.30	30.0	19.50
20.8	11.65	26.0	15.00	30.9	20.60
21.4	11.25	26.1	15.00	31.3	21.20
21.5	11.70	26.2	14.80	31.8	21.70
22.0	12.50	27.0	16.25	32.9	22.95
23.2	12.75	27.0	15.85		

TABLE I-A.- RELATIVE HUMIDITY PRODUCED BY CALCIUM CHLORIDE
SOLUTION OF SPECIFIC GRAVITY 1.30 AT 20°C.

Temperature, °C.	Vapour Pressure, of Water, mm.Hg.	*Vapour Pressure, CaCl ₂ Soln., mm.Hg.	Relative Humidity, %
14	11.9	7.2	60.5
15	12.7	7.6	59.8
16	13.5	8.1	60.0
17	14.4	8.6	59.7
18	15.3	9.2	60.2
19	16.3	9.8	60.1
20	17.4	10.4	59.7
21	18.5	11.1	60.0
22	19.6	11.8	60.2
23	20.9	12.7	60.8
24	22.2	13.5	60.8
25	23.5	14.4	61.3
26	25.0	15.2	60.8
27	26.5	16.2	61.1
28	28.1	17.2	61.2
29	29.7	18.3	61.6
30	31.5	19.5	61.9
31	33.4	20.7	62.0
32	35.3	22.0	62.3

* These values are taken from the curve, Figure 6.

Comments On The Method

It must not be thought that this method of air-drying coal, as distinguished from others, removes the surface moisture and that alone. It is thought, however, that no water of constitution is removed before the surface moisture has been given off. Some tests carried out at the Department of Mines, Ottawa, by E. Stansfield ⁽¹⁾ lend support to this. Surface moisture, then, as such, is not determined by the usual means of air-drying, standard or otherwise. Surface moisture and loss on air-drying are not the same thing, though they might be under some circumstances. Only the latter term represents a determinable quantity of moisture.

As before stated, this standardized method of air-drying gives "a measure of the water-retaining power of a coal." ⁽²⁾ While this section has dealt with loss in air-drying, the thing of importance was not the water lost, but the water retained. There are reasons for believing that under exactly similar conditions of air-drying similar coals would retain approximately the same percentage of moisture. The method of determining this moisture will be treated in the next section, but some results are given below in Tables II. and III., to indicate the success of this method of air-drying.

Entirely concordant results have not yet been obtained for high moisture coals; apparently slight differences in the drying conditions, and conditions inherent in the coal itself,

⁽¹⁾ Summary Report, Dept. of Mines, Ottawa, 1919, pp.38-39.

⁽²⁾ Trans. C.I.M. & M., Vol.XXVI., 1923, p.615.

such as, the inorganic matter and different proportions of the
banded constituents which make up the pure coal produce anomalies.⁽¹⁾

To test whether this last factor was of importance in Alberta coals, the following test was undertaken. A sample of clarain, or glance coal, and a sample of fusain, or "mineral charcoal" from the White Star Mine, Edmonton, were analysed for moisture and ash, with the following results:-

	<u>Air-Dry Basis</u>	
	<u>Moisture, %</u>	<u>Ash, %</u>
Clarain . .	18.16	3.22
Fusain . .	7.07	5.24

It can be seen, then, that two samples from the same mine having different proportions of these constituents will vary somewhat in the moisture they will retain under the same conditions of humidity. In spite, however, of this possibility, promising results have been obtained in the standard air-drying of high moisture coals. The experimental work with the standardized air-drying apparatus was carried out in connection with the routine work of coal analysis in the S. & I.R. C. Laboratory, and the figures quoted in the tables below are based on the routine work in the laboratory. Tables II. and III. show the retained moisture of a number of samples of coal from the same area, air-dried by this method of standard air-drying for a period of 48 hours. The coals were not air-dried on the same day, but over a period of several days, and in some cases after a lapse of several months,

(1) Chem. & Ind., Vol. 43, No. 18, p. 135T, May 2, 1924.

TABLE II.- AIR DRYING OF COAL (Drumheller Area, #1 Seam).

Sample No.	Total Moisture, %	Air-Dry Moisture, %	A s h, %	Variation from Mean
<u>LABORATORY</u>				
93-23	19.0	12.8	7.4	-0.5
94	17.7	11.7	6.3	-1.6
95	17.8	11.2	6.8	-2.1
96	17.8	11.3	7.1	-2.0
97	20.8	13.8	5.2	+0.5
98	19.8	13.1	7.0	-0.2
99	20.3	14.0	4.8	+0.7
100	20.5	13.6	5.3	+0.3
101	19.0	13.4	7.4	+0.1
102	18.4	15.2	7.8	+1.9
103	18.3	15.3	8.0	+2.0
104	18.3	14.5	7.8	+1.2
<u>STANDARD</u>				
155-23	18.8	14.3	7.9	-0.4 Nov.5
156	18.9	14.4	8.5	-0.3
157	18.5	14.4	7.6	-0.3
158	18.7	14.3	7.9	-0.4
159	18.1	14.4	4.7	-0.3
160	18.2	14.1	6.9	-0.6
161	18.4	14.4	6.8	-0.3
162	18.5	14.5	6.6	-0.2 Nov.22
42-24	17.0	14.2	6.7	-0.5 Mar.25
43	17.9	14.6	7.9	-0.1
44	18.0	14.3	8.7	-0.4
45	18.5	15.0	7.9	+0.3
46	19.0	15.2	6.1	+0.5
47	17.9	14.4	7.5	-0.3
48	18.3	15.0	7.5	+0.3
49	18.8	15.2	6.8	+0.5
58	20.5	15.2	8.7	+0.5 Apl.8
59	20.6	15.6	8.3	+0.9
60	21.6	15.1	10.4	+0.4
61	20.8	15.1	10.7	+0.4

Air-Dry Moisture	Lab.	St'd		Lab.	St'd
Maximum	15.3	15.6	Max.but one	15.2	15.2
Minimum	11.2	14.1	Min.but one	11.3	14.2
Difference	4.1	1.5		3.9	1.0
Average Moisture	13.3	14.7			
Average Variation from Mean				1.1	0.4

TABLE III.- AIR DRYING OF COAL (Carbon Area)

Sample No.	Total Moisture, %	Air-Dry Moisture, %	A s h, %	Variation from Mean
		<u>STANDARD 5 days</u>		
18-24	17.1	13.1	10.3	+0.1
19	17.5	13.5	10.3	+0.3
20	16.4	12.8	10.9	-0.4
21	16.4	13.0	10.2	-0.2
22	15.9	12.7	10.6	-0.5
23	16.8	13.3	8.4	+0.1
24	16.4	13.0	10.4	-0.2
25	16.9	13.6	8.9	+0.4
26	16.6	13.6	7.0	+0.4
27	16.5	13.7	8.1	+0.5
28	16.6	13.8	7.7	+0.6
29	16.4	13.6	7.3	+0.4
30	16.7	12.7	7.8	-0.5
31	16.0	13.2	9.0	0.0
32	16.4	13.2	9.4	0.0
33	15.9	13.2	9.5	0.0

Air-Dry Moisture

Maximum	13.8	Maximum but one	13.7
Minimum	<u>12.7</u>	Minimum but one	<u>12.8</u>
Difference	1.1		0.9
Average Moisture	13.2 %		
Average Variation from Mean	0.3 %		

so that the humidity in the laboratory was not the same when the coals were being air-dried. A comparison also is made between coals dried under ordinary laboratory conditions, and others from the same seam air-dried in the standardized way.

S u m m a r y

1. Air-drying loss is an arbitrary determination, and is not a determination of a definite constituent of the coal.

2. The Stansfield Constant Humidity Apparatus is a satisfactory standardized method for drying coal for the following reasons:-

(a) It maintains for all practical purposes a constant humidity, irrespective of laboratory conditions, and the atmosphere in the apparatus is maintained at approximately 60% relative humidity by the calcium chloride solution used of 1.30 specific gravity at ordinary laboratory temperatures.

(b) Similar coals from the same locality dried by this means approximate a percentage of moisture content which is characteristic of these coals.

In conclusion let it be said that by this method of air-drying the essential requirements for air-drying are met, and in addition much valuable information can be obtained regarding the moisture retentive power of coal. It tends to remove from a wet mine any stigma that might be attached to the quality of the coal due to excessive surface moisture. It has been adopted by the Department of Mines, Ottawa, and the Government of Alberta, as the standard method of air-drying. It has filled a marked need in the air-drying of Alberta coals which vary so widely in their moisture content, and offers a satisfactory means of overcoming the varying humidity conditions of the laboratory even in this Province, at different seasons of the year.

METHOD OF DETERMINATION OF RETAINED MOISTURE

Just as it was emphasized in the last section that surface moisture and loss on air-drying were not the same thing, so it should also be pointed out that moisture retained after air-drying and water of constitution are not the same thing, though they might be under some circumstances -- if, for example, a coal were dried in an atmosphere, the pressure of the water vapour of which was equal to the vapour pressure of the water of constitution in the coal. This makes an assumption which might be hard to prove, namely, that water of constitution has a definite vapour pressure. It has properties which point in that direction, in that moisture may be taken up or given up by a coal, depending on its condition with reference to the humidity of the atmosphere in which it is exposed.⁽¹⁾

H i s t o r i c a l

Methods of coal analysis have frequently been the subject of discussion and investigation. The widely varying results obtained by different methods have long been known; not the least variable of which have been those results obtained by the different methods of determining retained moisture.

In the year 1898 a committee was appointed by the American Chemical Society to report on methods of coal analysis. The

⁽¹⁾ "Hygroscopicity of some Canadian Fossil Fuels," G. Hoffman, Trans. Royal Soc. Canada, 1889, 1st Series, Vol. VII., Sec. 3.

committee consisted of W. T. Hillebrand, G. B. Dudley, and W. A. Noyes. The final report of this committee was published in 1899.⁽¹⁾ The committee's recommendation with reference to moisture was that 1 gram of powdered coal be dried at 104-107°C. for 1 hour, in a double-walled bath heated by boiling toluene. The dried coal was to be cooled in a desiccator and weighed. Sulphuric acid in a desiccator was also discussed as a means of determining the moisture in coal; it gave higher results, but was only recommended for very accurate work.

In 1906, E. E. Somermeier published an article dealing with the single topic, "Moisture in Coal," ⁽²⁾ in which he advocated determining the moisture by heating at 105°C. for 1 hour in a double-walled oven, a calcium chloride solution boiling at 105°C. being used for heating. He adds, however, that heated dry air should be forced through the oven during the determination.

Moisture in coal was made the subject of an international investigation in 1911. A sub-committee to the International Commission on Analysis was appointed in March of that year to go into this matter. The countries represented were England, France, Germany, Belgium, United States, Canada, Holland, Switzerland and Transvaal. The work of this committee was embodied in the report presented to the Eighth International Congress of Applied Chemistry held in New York in 1912.⁽³⁾ The Chairman of the committee was

⁽¹⁾ Journ. Am. Chem. Soc., Vol.21, 1899, pp.1116-1132.

⁽³⁾ No.10 sub-committee report by Messrs. Holloway & Coste, pp.77-128.

⁽²⁾ Jour. Am.Chem.Soc., Vol.XXVII, 1906, p. 1631.

G. T. Holloway, England, who, along with Coste, also of England, prepared the report for the Congress. E. Stansfield, then of the Department of Mines, Ottawa, now of the S. & I. R. C. A., was Canada's representative on that committee. ⁽¹⁾ Dr. Hillebrand, elsewhere mentioned, was the outstanding United States representative.

The work of the committee consisted of determining the moisture content of six samples of coal sent out from Great Britain, one of the six a sample from South America. The moisture content of the coal ranged from about 3% to 10%. Each member of the committee was asked to employ the following methods:-

- (1) 1 gram of coal heated to 105 to 107°C. for 1 hour.
- (2) " " " " " " " " " 2 hours.
- (3) The method in general use by such member of the committee.
- (4) Drying "in vacuo" over concentrated sulphuric acid.
- (5) The coal dried in a current of inert gas and the loss of weight determined. The moisture evolved to be absorbed in a calcium chloride tube and weighed.
- (6) The same procedure as in (5), but air used in place of an inert gas.

A few conclusions drawn from the reports received and work done are of interest:-

"Heating for 1 hour at 105 to 107°C. is sufficient. There appears to be no advantage in heating for a longer period."

"The method of drying at 105 to 107°C. in a current of gas free from oxygen, appears to possess a considerable advantage over any other process of drying at an elevated temperature, in that the necessary compromise between the

⁽¹⁾ The report of his work on the committee is contained in Sessional Paper No. 26A, Mines Branch, Canada, Summary Report, 1911, pp. 123-134.

desire to dry completely on the one hand, and the fear of oxidation on the other is reduced to a minimum."

"There is evidence that no appreciable amounts of hydrocarbons are lost by coal when it is exposed to temperatures not above, say, 140°C. for a time much longer than that required for drying it, although there is evidence that traces, which can be detected by suitable means, of gases are lost under certain circumstances."

In the fall of the same year, 1911, two committees were appointed in the United States to go into the matter of coal analysis. One was appointed by the American Society for Testing Materials, and the other by the American Chemical Society. In December these two committees formed a joint committee under the chairmanship of W. A. Noyes. Sub-committees from the joint committee were appointed consisting of two members and a chairman, each sub-committee being responsible for some phase of coal analysis. Methods of determination of moisture were assigned to the committee, under the chairmanship of Dr. Hillebrand. In June 1913 a preliminary report of this committee was published. (1)

It was recommended that powdered coal be heated to 105°C. for one hour; when anthracite or bituminous coals were being dried, a current of dry air (no volume stated) be passed through the oven, but for sub-bituminous and lignite coals, dry carbon dioxide or nitrogen be used in place of air.

In 1912 Stanton and Fieldner (2) in a paper, suggested that coal be heated to 105°C. and that dry air be passed through the oven at such a rate that the atmosphere be changed 8 to 10

(1) Jour. Ind. Eng. Chem., June 1913, Vol.V.

(2) A method of analysing coal and coke, F.M.Stanton and A.C. Fieldner, Tech. Paper No.8, U.S. Bureau of Mines.

times in 1 hour.

A second preliminary report of the joint committee mentioned above appeared in 1914, and the final report in the A.S.T.M. Year Book, 1915.(1)(2) The latest A.S.T.M. standards for moisture determination are essentially the same(3) as the second preliminary and final reports just referred to. The heating temperature is 104 to 110 ° C., and the atmosphere in the oven is changed at the rate of two to four times per minute by passing dry air through it.

The rate of change of gas or air is not looked upon as a factor in the determination of moisture in the early reports, though they agree with the later on the temperature at which moisture should be determined. There is a wide divergence between the rate suggested by Stanton and Fieldner and by the American Society for Testing Materials. The executive of the International Committee referred to above were most emphatic in their endorsement of the use of an inert gas in place of air as a drying agent, though no volume or rate of change was specified. With a view to finding out something definite about the rate of gas to be used, a series of tests were made. Air as a drying agent was excluded for the reason mentioned, namely oxidation, which is a matter of some importance in Alberta coals. Carbon dioxide was used at the beginning of the tests, but later natural gas was substituted.(4)

(1) Amer. Soc. for Testing Materials, Standards, Vol.XIV, 1914.

(2) Amer. Soc. for Testing Materials, Year Book, 1915.

(3) Amer. Soc. for Testing Materials, Standards, 1924.

(4) Alberta Natural Gas consists of 95% methane and 2 to 3% ethane. "Natural Gas in Canada", R.T.Ellworthy, Bulletin C.I.M. & M., No.155, March 1925, p.283.

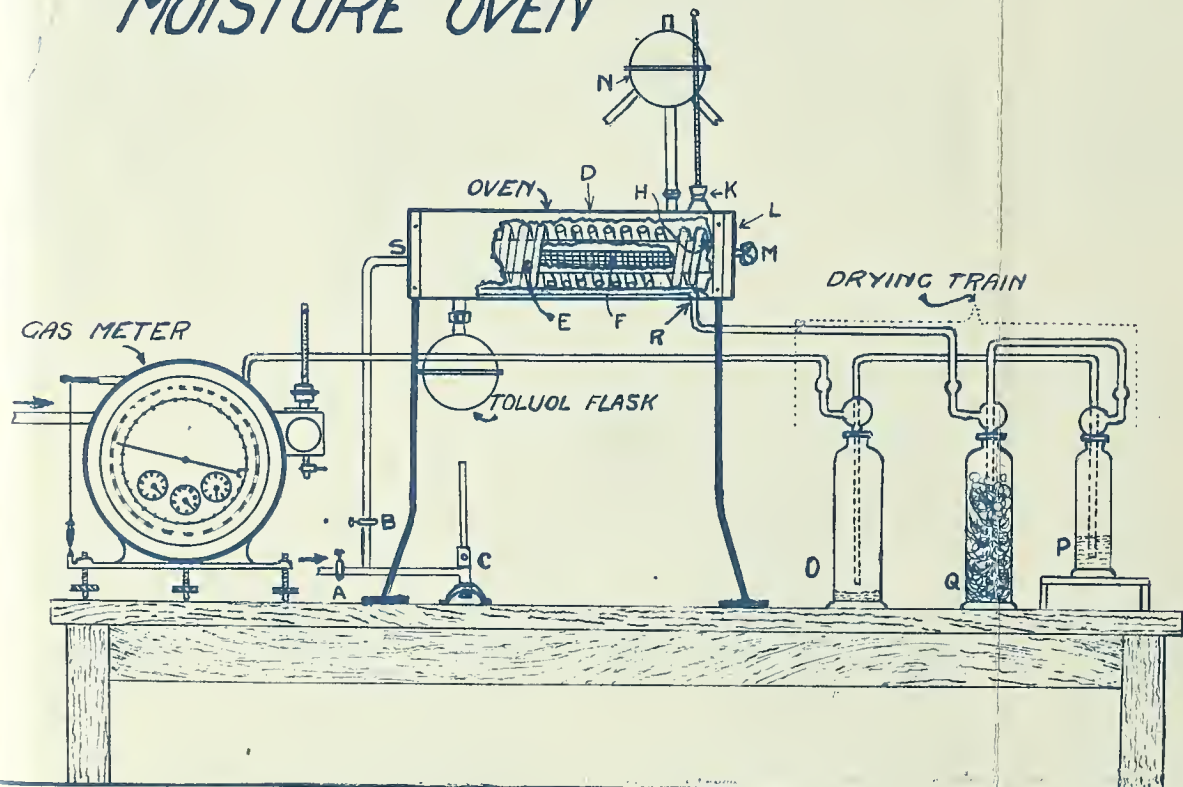
E x p e r i m e n t a l

The drying oven used was the type described by R. L. Sian⁽¹⁾ and manufactured by Baird & Tatlock (Figure 7, next page). The oven is 18" long by 3-5 /16" wide by 1-1/2" high inside. It consists of a copper jacketed chamber (E) containing a wire tray (F), a door fitted with a rubber gasket and secured by thumb screws (M,M). The oven is heated by means of the copper flask, containing toluol, the vapors of which envelope the chamber and enter the condenser at (N), returning to the copper flask. The drying gas is conducted to the jacket at (R) and passes through a worm, entering the oven at (H). Passing over the coal, the gas leaves at (S). A thermometer at (K) registers the temperature in the oven. The accessories shown in the figure (7) are those used with natural gas.

The gas from the mains enters the meter and passes to the drying train. A reversed drying bottle (O) with concentrated sulphuric acid in the bottom, the surface of which is clear of the tube leading to (P), acts as a combined trap and drying bottle, to protect the meter and at the same time dry the gas. The drying bottle (P) contains concentrated sulphuric acid for drying the gas, and (Q), glass wool, to prevent the mist of sulphuric acid being carried into the oven. On leaving the oven at (S), the natural gas is conducted to the bunsen burner and is used to heat the toluol. When carbon dioxide is used, the gas escapes into the fume cupboard. By a suitable adjustment of valves (A) and (B)

(¹) Jour.Soc.Chem.Ind., Vol.XXX, #2, Jan.31,1911, p.61.

MOISTURE OVEN





the gas may go direct to the burner or through the apparatus. Small unlacquered ointment tins, $1\frac{1}{2}'' \times \frac{1}{2}''$, with tightly fitting lids were used in making the moisture determinations. An Abbé ball mill was used for grinding samples, which were ground to pass 65-mesh, Tyler standard, opening 0.0082".

In making a moisture determination, the procedure is as follows: The pans are heated at a temperature of 110°C. for 30 minutes, then cooled in a desiccator. Approximately one gram of powdered coal is placed in the tared pan, covered, and weighed. Eight samples are usually weighed at a time. The pans are placed, with the lids beneath, in the oven tray, and pushed into the oven, which has previously been brought to 106°C.⁽¹⁾ As soon as the door is replaced, the gas is passed through the oven, and this treatment is continued for one hour, when the coals are removed, covered, placed in a desiccator, cooled "in vacuo" and weighed; the loss in weight being taken as the moisture content of the coal.

The method appears straight-forward, but it was discovered that for varying volumes of gas passed through the oven, varying results were obtained. (See Table IV., next page). Stanton and Fieldner, as before mentioned (page 34) had suggested 8 to 10 changes per hour, while the A.S.T.M. required that the air be renewed at the rate of 2 to 4 changes per minute. This means that in an oven of the size used (0.05 cu.ft. capacity) the former

⁽¹⁾ Toluol boils at 106°C. at this altitude.

Table IV.-

<u>Coal "A"</u>		<u>Coal "B"</u>		<u>Coal "C"</u>	
<u>Volume of Gas, cu.ft./hr.</u>	<u>Moisture, %</u>	<u>Volume of Gas, cu.ft./hr.</u>	<u>Moisture, %</u>	<u>Volume of Gas, cu.ft./hr.</u>	<u>Moisture, %</u>
0.42	9.95	0.55	17.75	0.57	0.74
0.86	10.29	0.77	17.83	0.91	0.74
0.92	10.34	1.08	17.94	1.11	0.77
1.31	10.45	1.29	18.07	1.47	0.76
1.60	10.54	1.55	18.09		
1.87	10.59	1.76	18.19		

requires that 1/2 cu.ft. of air pass through the oven per hour, while the latter requires 6 to 12 cu.ft. in the same time.

In addition to the uncertainty just mentioned, the variability of the amount of moisture given off by a coal with the volume of gas passed through the oven should be considered. This difficulty was also met. The loss in moisture was not uniform throughout the oven for low rates. The pans nearest the opening through which the gas entered the oven suffered the greatest loss in weight; this loss falling off, going from front to rear of the oven. (See Table V.) This latter effect was more or less to be

Table V.-

<u>Rate of Gas, cu.ft./hr.</u>	<u>% Moisture Removed</u>				
	<u>Rear</u>	<u>- -</u>	<u>to</u>	<u>- -</u>	<u>Front</u>
0.42	9.63	9.74	9.80	9.84	9.95
0.86	10.10	10.20	10.24	10.27	10.29
0.31	10.40	10.39	10.43	10.44	10.45

expected; as the drying power of the gas will diminish as it passes over the moist coal and takes up water. The matter, than, of a suitable rate at which to pass the gas through the oven, must be governed by the following conditions: (1) It must be such that there will be uniform drying in all parts of the oven; (2) it must give complete or maximum drying; (3) the rate must not be so fast that the gas cannot be heated to the required temperature, or that the risk is run of blowing coal out of the pans. Tests were carried out, keeping these conditions in mind, on coal of varying moisture content;

It was soon noticed that in order to make each run on a given coal comparable with other runs on that coal, the tests had to be made on the same day, as the lapse of a day or two would make an appreciable difference in the thermoisture content of the sample.

For example:-

<u>Date of Run</u>	<u>Gas Rate, cu.ft./hr</u>	<u>Moisture, %</u>	
		<u>Maximum.</u>	<u>Average of 5</u>
4 Jan.1924	2.76	7.07	<u>7.05</u>
7 Jan.1924	2.40	7.17	<u>Average of 4</u> 7.13

Other examples of the same effect could be given. Preliminary determinations were made, using carbon dioxide, to try to determine at what rate the highest value could be obtained for a given sample of coal. The results are shown below in Table VI. (next page) Evidently a rate in the neighborhood of 3.1 cu.ft. per hour appears to be about the rate required. This is slightly over one change per minute. Acting on this as a tentative rate for work

TABLE VI.- CARBON DIOXIDE AS A DRYING AGENT

Sample No.	Gas Rate, cu.ft./hr.	Moisture, %	
		Maximum	Average of 5 samples
128-23	2.74	18.40	18.34
" "	3.10	18.48	18.44
" "	3.28	18.44	18.39
7 -24	2.47	13.30	13.23
" "	3.00	13.40	13.32
" "	3.63	13.37	13.28
111-23	3.19	14.18	14.12
" "	3.24	14.11	14.05

in the routine analysis of the laboratory, a large number of samples were determined. The procedure adopted, in view of the fact that the drying might not be uniform throughout the oven, was to put the samples, usually eight in routine work, in the oven in a definite order, and in the check determination to reverse that order, so that the samples at the back in the first determination would be at the front in the duplicate. Table VII.(next page) gives the values of typical check determinations, the variations being compared with the A.S.T. M. Standard permissible differences.

With the installation of natural gas in the laboratory, it was decided to try it in place of carbon dioxide. Preliminary determinations were made with both gases, for comparison, on the same coals. The results are shown in Table VIII.

TABLE VII.- ROUTINE ANALYSIS RESULTS USING CARBON DIOXIDE

Batch No.	Determination		Difference
	1	2	
1	1.16	1.12	0.04
	1.06	1.07	0.01
	1.04	1.06	0.02
	0.99	1.06	0.07
	1.16	1.15	0.01
	1.06	1.03	0.03
	19.49	19.48	0.01
	18.57	18.65	0.08
	18.69	18.88	0.19
2	11.10	11.21	0.11
	5.22	5.22	0.00
	12.78	12.76	0.02
	13.16	13.31	0.15
	7.80	7.76	0.04
	8.11	8.09	0.02
	8.23	8.28	0.05
	8.52	8.53	0.01
3	15.27	15.34	0.07
	14.67	14.62	0.05
	10.58	10.50	0.08
	10.54	10.62	0.08
	10.73	10.63	0.10
	11.45	10.55	0.10
	11.65	11.62	0.03
	14.03	13.96	0.07
	14.14	13.97	0.17
	17.99	17.87	0.12
4	18.58	18.58	0.00
	18.14	18.24	0.10
	17.75	17.75	0.00
	17.83	17.98	0.15

Note 1.- The above are typical runs taken from laboratory notebook with a view to having a complete series of moisture contents.

Note 2.- The A.S.T.M. Standard permissible difference is 0.2 where the moisture is less than 5%, and 0.3 where the moisture is over 5%. Differences of over 0.1 are regarded as unsatisfactory in Alberta work.

TABLE VIII.- CARBON DIOXIDE AND NATURAL GAS COMPARED

Sample No.	Gas Rate, cu.ft./hr.	M o i s t u r e , %	
		Maximum	Average of 5 runs
	<u>CO₂</u>		
111-23	3.19	14.18	14.10
" "	3.24	14.11	14.05
	<u>Natural Gas</u>		
111-23	2.91	14.07	14.03
" "	3.03	14.15	14.11

The results being satisfactory, a series of tests were made using natural gas at different rates. It appeared to be more erratic in its action than carbon dioxide, but a rate in the neighborhood of 3.5 cu.ft. per hour gave as high results as any for most of the coals. (Table IX.)

TABLE IX.- DRYING WITH NATURAL GAS

Sample No.	Gas Rate, cu.ft./hr.	M o i s t u r e , %	
		Maximum	Average of 5 runs
25-25	2.77	0.99	0.95
" "	3.65	1.00	0.96
" "	4.55	1.00	0.98
" "	4.94	1.00	0.99
68-25	3.57	7.75	7.72
" "	3.76	7.72	7.67
" "	4.88	7.75	7.73
71-24	2.95	11.62	11.60
" "	4.28	11.63	11.59
" "	5.09	11.77	11.70
125-24	3.47	19.22	19.14
" "	4.19	19.10	19.06
" "	5.23	19.15	19.09
6-25	3.36	14.82	14.71
" "	4.73	14.72	14.68
" "	6.11	14.85	14.78

The procedure, then, when using natural gas for routine analysis was the same as before, but the rate was changed to 3.5 cu.ft. per hour in the initial run and in the duplicate the rate was increased to 5 cu.ft. per hour. The results obtained appear to justify the choice of rates. (Table X., next page).

During the course of the tests, just described, the question was raised as to the temperature of the gas entering the drying oven. Did the rate at which the gas passed through the oven make any difference in the temperature of the gas? To settle the point, the following experiment was carried out. The door of the oven was replaced by a piece of asbestos board of similar size and with a suitable gasket. It was bored so as to permit a thermometer being passed through it; the bulb, being at the point where the gas entered the oven, would register the temperature of the gas entering. The thermometer usually in the oven was left there, so that comparisons could be made. The oven was brought to the temperature desired, and the gas was passed through at various rates, the rate being maintained constant till no further change in the thermometer was noted. Ten minutes was found to be ample time. It was observed that the thermometer at the orifice, before the passage of gas, registered a lower temperature than the other. This condition was changed, however, as soon as the passage of the gas began. Table XI. shows the results obtained in order of determination. (Page 46) (A) is the thermometer at the usual point (See Figure 7, page 37), and (B) is the thermometer at the gas orifice.

TABLE X.- ROUTINE ANALYSIS RESULTS USING NATURAL GAS

Batch No.	Determination		Difference
	1	2	
1	13.66	13.69	0.03
	15.71	15.72	0.01
	15.51	15.61	0.10
	15.21	15.31	0.10
	17.17	17.18	0.01
	16.94	17.01	0.07
	17.55	17.55	0.00
	16.59	15.67	0.08
2	7.41	7.41	0.00
	7.54	7.51	0.03
	8.63	8.61	0.02
	12.68	12.66	0.02
3	0.46	0.44	0.02
	0.50	0.55	0.05
	2.46	2.58	0.12*
	1.33	1.33	0.00
	3.72	3.70	0.02
	3.80	3.89	0.09
	0.75	0.72	0.03
	0.70	0.62	0.05
	0.60	0.56	0.04
	12.43	12.28	0.15*
4	11.87	11.87	0.00
	11.23	11.31	0.08
	12.61	12.78	0.17*
	12.40	12.42	0.02
	12.63	12.72	0.09
	12.92	12.92	0.00
	13.07	13.03	0.04
	0.42	0.42	0.00
5	18.03	18.05	0.02
	17.56	17.54	0.02
	17.75	17.76	0.01
	17.84	17.68	0.16*
	17.67	17.57	0.10
	18.15	18.05	0.10

Note 1.- The above are typical runs taken from laboratory notebook with a view to having a complete series of moisture contents.

Note 2.- The A.S.T.M. Standard permissible difference is 0.2 where the moisture is less than 5%, and 0.3 where the moisture is over 5%. Differences of over 0.1 are regarded as unsatisfactory in Alberta work. Samples marked * are the only ones which do not measure up to the more rigid standard.

TABLE XI.- TEMPERATURES USING DIFFERENT RATES OF DRYING GAS IN OVEN

A	B	CO ₂ Rate, cu.ft./hr.	A	B	CO ₂ Rate, cu.ft./hr.
106.8°	105.0°	0.0	106.4°	105.9°	5.2
106.8°	105.8°	2.2	106.3°	105.7°	2.1
106.6°	105.9°	3.2	106.3°	105.9°	3.9
106.5°	106.0°	7.9	106.3°	105.0°	8.3
106.4°	106.0°	4.7	106.3°	104.7°	0.0
106.4°	105.9°	3.3	106.3°	104.5°	0.0

Note.- The lowering of the temperature in the toluol oven has no significance. This was due to a lowering of the temperature in the fume cupboard during the course of the test.

It is evident from the results that so far as temperature is concerned, a fairly fast rate is the most satisfactory. Other considerations of course enter in; such possibilities as insufficient heating of gas and mechanical loss.

A further problem related to determination of moisture in coal was taken up, namely:-

Loss of Hydrocarbons at 106°C.

Analysis for moisture by the method just outlined appeared to some operators in the Province to give high results, and it was suggested that possibly hydrocarbons were being driven off along with the moisture. Much has been done and written on this topic, but it was thought advisable to establish that what held for coal elsewhere, namely that the loss was negligible, was

also true of Alberta coal. For this reason the following test was undertaken. Four samples of the same coal were weighed out in the usual manner and placed in a desiccator over concentrated sulphuric acid. The desiccator was exhausted, then filled with natural gas; this was repeated three times, and the desiccator was finally exhausted. The desiccator was set aside for 14 days when the samples were weighed, and the moisture loss determined. The samples were then placed in the toluol oven for one hour at 105°C., and the loss again determined. The following are the results:-

Average of 4 Samples

Loss in Desiccator	10.97 %
Loss at 105°C.	0.52 %

If we assume that all the water was removed by the sulphuric acid, then 0.52% was the loss in volatile matter, but later evidence points to the fact that the value 0.52% was not all volatile matter; for, drying samples of the same coal for 22 days, the loss at 105°C. was reduced to 0.35%. Longer drying would probably have reduced it still further. This all goes to show that volatile matter losses at 105°C. are small, which bears out the findings of the International Committee already mentioned on page 17, and of Dr. Hillebrand, who shows the loss in volatile matter at 105°C. for coals which he tested to be less than 0.1%.⁽¹⁾

⁽¹⁾ Jour. Amer. Chem. Soc., Vol.20, 1898.

S u m m a r y

(1) A method of determining moisture in coal is described, using carbon dioxide or natural gas. For carbon dioxide, the atmosphere in the drying oven should be changed at the rate of 62 changes per hour. For natural gas, the rate should be equal to 70 changes per hour for the first determination and 100 for the duplicate.

(2) By the use of this method a stricter standard may be set up than A.S.T. M. permissible differences in duplicate determinations ⁽²⁾. The following scale of permissible differences is suggested for Alberta coal:-

<u>T % Moisture in Air-dried Coal</u>	<u>Permissible Difference</u>
0.0 to 5.0	0.10
5.0 to 10.0	0.15
10.0 to 20.0	0.20

(3) The loss of volatile matter in determining moisture by this means is negligible.

DISTRIBUTION OF MOISTURE IN RELATION TO ASH

The ash, or, more correctly, the mineral matter in coal appears to bear some relation to the moisture; it has been noticed that if, for example, two samples of the same coal, with the same total moisture are air-dried under the same conditions, the coal

(2) Amer. Soc. Test. Mat., 1924, p.

with the higher ash tends to retain less water than the other, though there are frequent exceptions to this. Over a small area the moisture content of coals in that area falls off as the ash increases. This seems to hold for high moisture coals, but for very low moisture coals this might not be the case.

With a view to getting some information about the relation of moisture and ash in coal, the following test was undertaken. A coal of known moisture content was separated into fractions of varying ash content by means of heavy solutions ⁽¹⁾. Carbon tetrachloride and benzol were used. The separated portions were exposed in the Stansfield Constant Humidity Apparatus for 48 hours and were then ground and analysed for ash and moisture. Three coals were treated in this way, varying in original moisture content from about 12 to 0.5%. The values of ash and moisture obtained in the separated fractions were plotted against each other. Their relation appears to be a linear one. (Graph, Figure 8, and Table XII., next page.) It would be unwise at the present time to express mathematically the moisture as a function of ash for any coal tested and expect that this relation would hold rigidly for other samples of the same coal. For different drying conditions, different results would be obtained as the upper line clearly shows. This much is certain, that if the coals are all dried under the same conditions, the curves represent the relative moisture retaining properties of the coal. Since the coals shown, except the upper curve, were

(1) Third Annual Report, S. & I.R.C. Alberta, 1922, p.29.

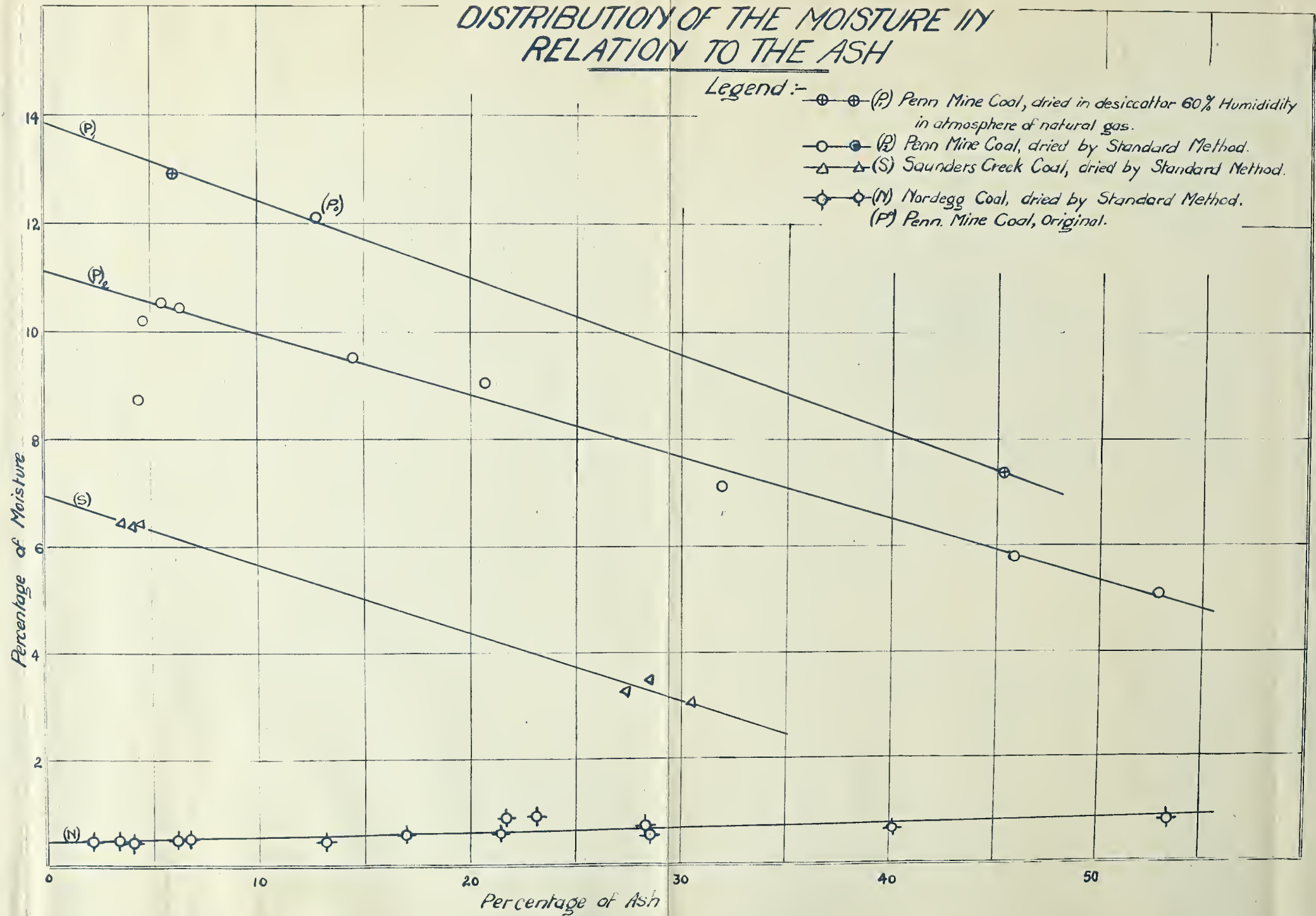
TABLE XII.- MOISTURE IN RELATION TO ASH

Coal	Ash, %	Moisture, %	Coal	Ash, %	Moisture, %
Saunders	9.45	4.13	Penn Mine	4.46	8.74
	4.49	6.41		14.51	9.54
	28.51	3.52		4.67	10.21
	30.52	3.04		31.86	7.14
	3.66	6.44		5.51	10.57
	22.22	4.54		45.84	5.79
Brazeau	2.09	0.45		6.44	10.44
	3.38	0.47		52.80	5.08
	40.21	0.63		12.85	12.10
	53.52	0.81		4.88	11.89
	13.22	0.41		35.22	7.65
	28.31	0.71		5.97	12.92*
	21.59	0.88		45.27	7.38*
	23.08	0.94		3.46	10.92
	21.35	0.58		12.94	10.41
	4.07	0.44		4.52	11.65
	28.64	0.56		32.48	7.65
	6.74	0.51		20.55	9.55
	16.88	0.57		* Dried in desiccator.	
	6.12	0.45			
	28.54	0.54			

DISTRIBUTION OF THE MOISTURE IN RELATION TO THE ASH

Legend :-

- ⊕ ⊕ (P) Penn Mine Coal, dried in desiccator 60% Humidity in atmosphere of natural gas.
- ● (P₂) Penn Mine Coal, dried by Standard Method.
- △ △ (S) Saunders Creek Coal, dried by Standard Method.
- ⊙ ⊙ (N) Nordegg Coal, dried by Standard Method.
- (P) Penn. Mine Coal, Original.



dried under similar conditions, the relation shown has significance. The uppermost curve is of interest in that the portions determining it were dried under slightly different conditions from the others. These two portions were kept in a desiccator filled with natural gas for 58 hours over a solution of calcium chloride of 1.30 Sp.Gr.. It is worthy of notice that the line joining them passes through the value for the original coal. The second curve from the top is for the same coal as the upper one, but the different drying conditions gave it values lower than the original coal. It would appear that this latter method offers a very convenient way of comparing the moisture retaining power of a coal, the conditions being so readily duplicated. For lack of time, further tests could not be undertaken along this line. Before leaving this topic, it should be noted that the Nordegg coal shows a slight increase in moisture with increase in ash. Why this is so is difficult to say, but it would appear from the nature of the powdered coal, which can only be wet with difficulty, that the inorganic constituents of this particular coal have a greater capacity for retaining moisture than the pure coal substance.

Summary.- (1) There appears to be a tendency in air-dried lignites and sub-bituminous coals for the low ash sample to retain more moisture than the high ash. In bituminous coals the moisture is practically uniform, but it has been found to increase slightly with increase of ash.

(2) The relation of moisture to ash in portions of coal

separated by heavy solutions, appears to be linear, but does not admit of mathematical treatment, as the moisture changes are too sensitive to slight changes in air-drying conditions.

3. A heavy solution method is described for determining the moisture retentive power of coal for any given conditions of humidity.

WATER OF OXIDATION

Reference was made earlier to a fourth type of moisture, namely, that which forms in coal. "Water of Oxidation" has been suggested as a name, since it was thought that it was a product of oxidation of coal. J. W. Lewis ⁽¹⁾ had pointed out that the moisture in ground coal did increase, but did not suggest a reason. This fact set forth by Lewis was made the subject of further investigation, two methods being adopted in dealing with it.

Experimental

For a period of over a year, a sample of fines was taken every Monday morning from the University power house bins. The sample was riffled in two equal parts; one part was analysed immediately for water content, the other part was weighed in a tared beaker, 600 cc. capacity, and was exposed for four weeks in an open shed, at the end of which time the moisture content was determined. The primary objects of this test were to find out how the total moisture changed from week to week in a coal from

*All coal that passes through a screen with 1/2" openings.

the same mine, and to obtain some information concerning the variations in loss of moisture of the exposed coal, for different seasons of the year. Incidental to the above, and this is the part which is related to the topic under discussion, use was made of this test to determine, if possible, whether or not water was formed in the coal during the period of exposure. It was thought that if water were formed in the coal, it could be accounted for by the loss of weight in the coal and the moisture retained. Another part related to the topic, also the outcome of the test, consisted of re-analysing the ground samples (the samples having been kept from the beginning of the test, 65 weeks) for moisture, to check up any charges. The first part of the test gave no conclusive results as to the formation of moisture in the exposed coal, but the results favour the assumption, as 37 tests out of 65 showed an increase averaging 0.4%, 22 tests showed a decrease averaging 0.35%, and 4 tests showed no change, 2 tests being rejected.

The results obtained for the second part of Method 1 were conclusive, and pointed unmistakably to the fact that in ground coal stored under conditions of the test, moisture is formed. Before giving figures it should be stated that where badly fitting stoppers were found the coal showed no increase in moisture and in some cases there was actually a loss in moisture. In cases where the stopper was faulty, the results have been excluded from the averages given below. The highest increase obtained on the re-determined moisture in the original coal was 1.2% while on the exposed coal the highest increase was 1.1%. On the average,

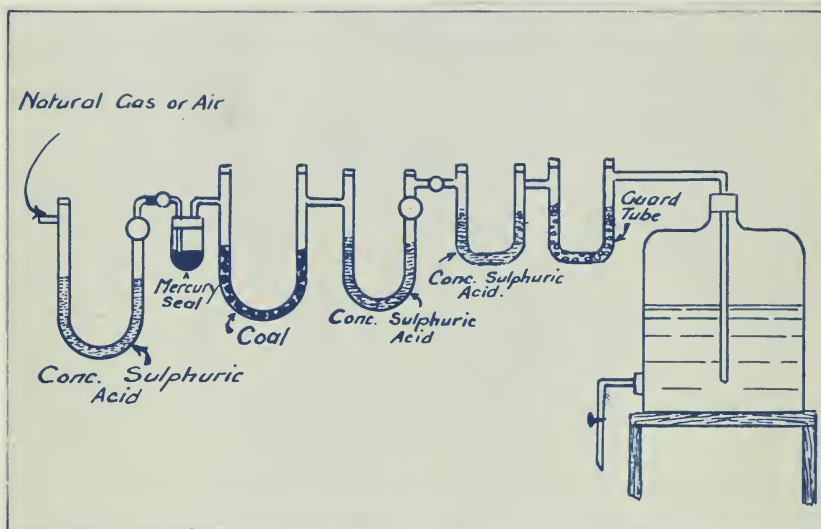
however, the exposed coal gave the higher results. Table XIII. shows the averages over periods of three months approximately of weekly test.

TABLE XIII.- STORED COAL TESTS

Period of Storage, months	Moisture Increase During Storage, %	
	Original Coal	Exposed Coal
12 - 16	0.7	0.5
9 - 12	0.3	0.4
6 - 9	0.2	0.6
3 - 6	0.3	0.3
1 - 3	0.2	0.2

The second Method by which the problem was dealt with, while of shorter duration in point of time than the other, gave decisive results. A sample of fresh coal from the same source as that used in the previous test was treated as follows:- One part was analysed for moisture and calorific value; another part of the same sample (ground to pass 8-mesh and retained on 14-mesh) was placed in a U-tube. This tube was then connected up with a drying and ^{an} air absorbing train in the manner shown in Figure 9 (next page). A definite volume of dry natural gas was aspirated through this apparatus; the moisture removed from the coal, and the gain in weight in the sulphuric acid were checked up every day. A similar tube filled with a sample of the same coal was connected up in the same manner, only dry laboratory air was aspirated through the

Fig. 9.



apparatus in place of natural gas. This test was run for a little over a month. During the test it was noticed that where natural gas was used the loss in weight of the coal checked up very closely with the gain in weight of the sulphuric acid. Only towards the end of the test did the sulphuric acid show a slight increase in weight over the loss in weight of the coal. In the case of the other sample where air was used, the loss in weight of the coal was uniformly less than the gain in weight of the sulphuric acid, and the final result showed quite a wide discrepancy. At the end of the test each coal sample was analysed for moisture and calorific value, and these values were checked up with the values of the original sample. Table XIV. shows the results obtained.

TABLE XIV.- MOISTURE FORMATION BY ACTION OF AIR AT ROOM TEMPERATURES

	Dried in Air	Dried in Natural Gas
(x) Moisture in original coal.....%	26.35%	26.35%
Calorific Value of " " (dry)B.T.U. per pound	11,334	11,334
Volume of gas passed through..cu.ft.	8.56	8.65
(a) Loss in weight of coal.....g.	22.01	19.66
(b) Gain in weight of H ₂ SO ₄g.	25.37	20.06
(c) Moisture in coal at end of test..... % of original coal	3.88	6.43
(d) Moisture determined from losses in weight (a) + (c).....g.	25.89	26.09
(e) Moisture determined from gain in weight of H ₂ SO ₄ (b) + (c).g.	29.25	26.49
"Water of Oxidation" (e) - (x)...g.	2.90	0.14
Calorific Value of dry coal after test...B.T.U. per pound	11,180	11,251
Loss in Calorific Value during test..B.T.U. per pound	154	83

It is to be seen from the table that the value for loss in moisture of the coal treated with air is a fair check on the value for the original coal. It must be remembered, however, that the value obtained does not represent the loss in moisture alone, but is the resultant of two processes having opposite effects, viz:- oxidation which increases the weight of the coal, and loss of moisture which decreases the weight.

It should also be noted that there is a decrease in the calorific value of the treated coal, the air treated coal suffering the greater loss.

Several important conclusions may be drawn from these tests just described. From the results obtained in the first test it can be definitely stated that:-

(1) Water is formed in ground lignite even when kept in glass bottles with ground-in stoppers.

(2) There is not yet enough evidence to state definitely that moisture is formed in lump coal exposed to the atmosphere, but indirect evidence (1) lends support to the view that moisture is formed.

The first finding has an important bearing on ultimate and proximate analysis of coal, and on the determination of the calorific value. It shows that the analysis should be carried out very soon after the coal has been ground; and if for any reason re-determinations of calorific value or constituents of coal other

(1) "Analyses of Canadian Fuels", E.Stansfield & J.H.H.Nicolls, Bulletin 25, Dept. of Mines, pp.43 & 58.

than moisture, have to be made after a lapse of a few days, the moisture should be determined in order to calculate back to the original moisture content, so that a check on the original determination can be obtained.

TABLE XV.- CHANGES IN ANALYSIS IN ANALYST'S SAMPLES

Date	Days Elapsed	Moisture %	Calorific Value, B.T.U.per lb.	
			As Determined	Dry Basis
Mar.14	13	13.59	10,121	11,711
27		13.90	10,080	11,707
Change..		+0.31	- 41	- 4
Mar.13	18	13.14	10,072	11,595
" 31		13.43	10,035	11,591
Change..		+0.29	- 37	- 4
Apl.10	30	14.82	9,682	11,366
May 10		15.25	9,626	11,357
Change..		+0.43	- 56	- 9
Apl.10	32	15.43	9,865	11,665
May 12		16.07	9,803	11,680
Change..		+0.64	- 62	+ 15
Mar.28	43	14.42	9,994	11,678
May 10		14.79	9,896	11,613
Change..		+0.37	- 98	- 65
Average Change in Calorific Value.....			- 60	- 20

In the table above it should be noted that moisture changes in the coal are clearly brought out; and that the drop in calorific value as determined is quite marked, but when calculated to a dry basis the discrepancy is practically negligible in the shorter periods.

The results of the second test have an important bearing on storage of coal, but since that is outside the scope of this thesis, it will not be dealt with. One important conclusion may be drawn which has a bearing on the determination of retained moisture, namely that natural gas as an inert drying agent in the determination of moisture is admirably suited for the work, as it does not appear to have any significant effect on the coal, even after exposure to it for one month. Finally, it would appear that "Water of Oxidation" is the product of the action of atmospheric oxygen on the coal, and not a product of auto-oxidation in the coal.

CHAPTER II.

MINERAL MATTER IN COAL

INTRODUCTION

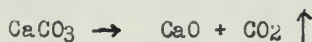
Mineral matter in coal is derived from two sources:-

- (1) The mineral matter which has formed an integral part of the vegetation from which the coal has been derived.
- (2) The mineral matter laid down along with the vegetation which formed the seam, and perhaps from infiltrations of mineral matter into the seam at a later period.

Mineral Constituents.- The compounds which go to make up the mineral matter, commonly occurring in coal are:- pyrites, marcasite; sulphates of iron, and calcium; carbonates of iron, calcium, and magnesium; and clays, shales, and gypsum. These compounds are not found in the same proportions in every coal, but vary both in kind and quantity.

Mineral Matter in Relation to Ash.- This mineral matter is the source of the ash which remains when coal is ignited. The ash, however, does not represent all the inccombustible material which is in the crude coal, but only the material which is non-combustible and non-volatile at the temperatures at which ash is produced. In the process of combustion, compounds have been

driven off which form a part of the mineral matter in the coal as mined, and are as inert as the material of which the ash consists. From pyrites, on combustion, inert matter is lost in the form of sulphur dioxide; from the sulphates, sulphur trioxide; carbon dioxide from the carbonates; and water of hydration from the clays, shales, and gypsum; which means that only part of the mineral matter in the coal is found in the ash. For example, if calcium carbonate occurs in the crude coal, only calcium oxide will be found in the ash, the decomposition taking place according to the equation:-



which means that 100 parts of calcium carbonate in the coal is represented by 56 parts of calcium oxide in the ash. With pyrites the reaction is more involved:-



in this case 240 parts of pyrites in the coal are represented by 160 parts of ferric oxide in the ash.

Mineral matter, then, in coal, from the point of view of the ash as determined, is comprised of the ash plus the volatile inert compounds which are given off. The value obtained by correcting the ash for volatile matter lost is commonly named "corrected ash". The term is a misnomer in that the ash as determined is the ash of the coal. While it is true that the ash is inert matter, it is not true that the volatile inert matter is ash. Strictly speaking, the term used should not be "corrected ash", but "ash corrected to inorganic, non-coal, or mineral matter." In future, when reference

is made to corrected ash, the term "corrected to mineral matter" will be used.

Determination of Ash.- In proximate analysis, ash is one of the constituents for which determination is made. The standard of the American Society for Testing Materials ⁽¹⁾ requires that the ash in coal be determined by heating 1 gram of coal in a crucible in a muffle furnace or over a bunsen burner. The temperature at which the determination is made should be between 700 and 750°C.. The coal is heated till the change in weight is not greater than ± 0.001 gram. The ash is cooled in a desiccator and weighed. In all the ash determinations carried out in connection with the work, later described, the above standard was followed.

HISTORICAL

The fact has long been known that volatile inert matter is driven off when the mineral impurities in coal are ignited. It was thought for some time that only sulphur was lost in the determination of ash. S.W.Parr ⁽²⁾ in 1906 pointed out that there were other compounds besides sulphur lost in the determination of ash. Two years later, 1908, W.F. Wheeler ⁽³⁾ published analytical evidence in support of the idea. In February, 1909, W. Brinsmaid published an article ⁽⁴⁾ on "The Amount of Inert Volatile Matter in the Mineral Constituents of Coal." In this article the writer pointed out that

⁽¹⁾ A.S.T.M., 1924, p. 987

⁽²⁾ "Unit Coal and the Composition of Coal Ash", S.W. Parr & W.F. Wheeler, Univ. of Ill., Bull.37, p.5.

⁽³⁾ Trans. Am.Inst.Min.Eng., Vol.38, p.621, 1908.

⁽⁴⁾ Jour. Ind.Eng.Chem. Vol.I., Feb.1909, p.65.

the non-volatile matter, weighed as ash, did not represent the weight of the inorganic matter in the coal. He described a method by which the amount of inert matter lost could be estimated. It consisted of taking two samples of the same coal, one with low ash and the other with high. The samples were ground separately to pass 100-mesh, and then varying proportions of them were taken, making a number of samples having a regular gradation of ash content. For example, if the original samples had ash contents of 5 and 20%, from them samples were made up having 8, 11, 14 and 17%. Determinations for ash and calorific values were made on these samples; the values so obtained were plotted, ash against calorific value. He did not indicate whether or not his results were calculated to a dry basis. It was found that the values plotted lay along a straight line. He produced the line to cut the horizontal or calorific value axis; the point of intersection he called the calorific value of the pure or ash-free coal. He then calculated from the graph what drop in calorific value an increase of 1% ash represented. It was seen, using the value obtained, that the available heat was used up before 100% ash was reached. That is to say, the straight line, determined above, when produced, meets the vertical or ash axis at a point of less than 100% ash for zero calorific value. He pointed out then that what is weighed as ash did not represent the total inert matter in the coal, but by joining the point of calorific value of ash free coal and the point for 100% ash and zero calorific value the drop in calorific value for 1% "inert matter" could be obtained. In order to correct the ash for the volatile inert

matter lost, he multiplied the ash as determined by a factor which he obtained by dividing 100 by the value of the point where the line determined cut the vertical or ash axis, or axis of zero calorific value.

In August of the same year, 1909, S.W. Parr and W.F. Wheeler, of the University of Illinois, published the bulletin already referred to, which deals with the same topic, but in a more direct way. They pointed out that the calorific value of the actual or pure coal substance for a given seam was practically uniform, and that its determination hinged on the correct determination of the inorganic constituents of the coal. They define "unit coal" as follows: "By unit coal is meant the organic material which is involved in combustion as apart from the mineral constituents which are extraneous and variable accompaniments of the actual or unit coal." They use the term "unit coal" in the sense in which the words "ash free" or "pure coal" are used above. They develop formulae for calculating the calorific value of "unit coal" which take into account all the non-coal constituents, i.e., ash and volatile inert matter. The non-coal constituents, they considered to be pyrites, the carbonates, chlorides, and water of hydration.

In 1921 Illingworth ⁽¹⁾ also pointed out that the total inert matter in coal did not consist of the ash as determined, but was the sum of the following constituents:-

(¹) "Analysis of Coal and its By-Products", S. Roy Illingworth, p.84.

- "(a) The total ash, as determined, less $\frac{4}{3}$ (¹) of the pyrites present.
- (b) The pyrites present.
- (c) The sulphates present in the coal calculated as SO₃ less the sulphates present in the ash.
- (d) The total water arising from the coal, i.e., the moisture plus the water in the form of inert volatile matter.
- (e) The total carbon dioxide occurring in the coal as carbonates, occluded, and in the form of inert volatile matter."

He outlined methods for the determination of the constituents named above except water of hydration.

In 1922 J.W. Lewis (²) in the laboratory of the Scientific & Industrial Research Council of Alberta developed a heavy solution method for determining the calorific value of pure coal and the measure of the loss of mineral matter incident to the determination of the ash. The method is as follows:- The coal sample was separated into two parts by means of heavy solutions in a specially constructed cup, the separation being accelerated by centrifuging. Solutions of calcium chloride or calcium nitrate of suitable specific gravity were used, the float being largely clean coal and the sink bone coal, depending on the specific gravity of the solution. By using solutions of different specific gravities, portions of widely varying ash content could be obtained. These portions were analysed

(¹) This is obviously an error. It should read " $\frac{2}{3}$ of the pyrites present."

(²) Third Annual Report, S. & I. R. C. A., pp.29-31.

for ash, moisture, and calorific value; the values for ash and calorific value were calculated to a dry basis and plotted against calorific value. The points so plotted lay on a straight line.

The results obtained resemble the Brinsmaid results, but the methods differ in the treatment of the samples. By the Brinsmaid method only a straight line could be obtained, since different proportions of the same two samples were taken. In the method just described a single sample was taken. A much wider range of ash content can be obtained by this method, so that the direction of the line is more clearly defined. As in the Brinsmaid method, the points of interest are the calorific value of the pure coal, and the point where the produced line cuts the ash axis at zero calorific value.

Several heavy solution curves of Alberta coal are included in the report mentioned, and since that time others have been determined. All of them cut the ash axis at points lying between 78.5 and 95.5%, which means that in determining the ash a loss of from 21.5 to 4.5% of its weight is lost in the form of volatile mineral matter.

EXPERIMENTAL

With a view to finding out something about these volatile compounds and the extent to which they occur in some Alberta coals, the following experimental work was undertaken:

- (1) Determination of the loss of volatile inert matter by heavy solution curves.
- (2) The quantitative determination, by analysis, where possible, of the volatile inert matter given off by the mineral constituents.
- (3) The determination of the mineral matter by extraction with hydrochloric and hydrofluoric acid.

The method required that a heavy solution curve be determined for each coal tested; that a part of the same sample be separated by a heavy solution of such specific gravity that the sink would have a high ash content; that on this sink, analyses be made for pyritic sulphur, sulphur as sulphates, and carbon dioxide as carbonates; and that a part of the sink be extracted by the acids mentioned.

Preparation of Samples.

Heavy Solution Curves:- Departures were made from the procedure outlined for determining heavy solution curves. In place of the calcium nitrate and calcium chloride solutions, solutions of carbon tetrachloride and benzene were used. By this change the work could be more expeditiously carried out in making the separation, washing the separated portions free of calcium salts, and in drying the coal after separation. A change was also made in the method of drying the coal before and after treatment. The sample was first exposed in the "Stansfield Constant Humidity Apparatus" for 48 hours, then, after treatment with the heavy solutions was again exposed in the apparatus. It was hoped that in so doing the moisture of the separated parts, float and sink, would together equal that of the original coal. It had been noticed previously that the parts lost moisture, and their combined moisture was less than the moisture of the original sample. Much improved results were obtained by these changes

Quantitative Analyses:- The fraction

selected for analysis for inert volatile matter, was obtained by filling a cylinder with the solution of the required specific gravity and then stirring in the coal, which had been ground fine. The coal was allowed to settle. After settling, the clean coal on the top was skimmed off and more coal was added with stirring and allowed to settle. This was repeated until 80 to 100 grams of sink were obtained, which was sufficient for the analyses.

Coals Tested:- In selecting coals for this work it was desired that these be as representative as possible. A sample was selected from each of the three coal bearing horizons in the province, viz:- Kootenay, Belly River and Edmonton.⁽¹⁾

Saunder's Creek Coal.

The coal first treated was from the Saunder's Creek area, a sub-bituminous coal, from the Belly River Horizon. There were two reasons for dealing with it first. (1) The heavy solution curve indicated a high loss of volatile inert matter in the determination of ash, and (2) it had a medium moisture content, normally around 7 or 8 %. The latter assured some

⁽¹⁾ Fourth Annual Report S. & I. R. C. A.

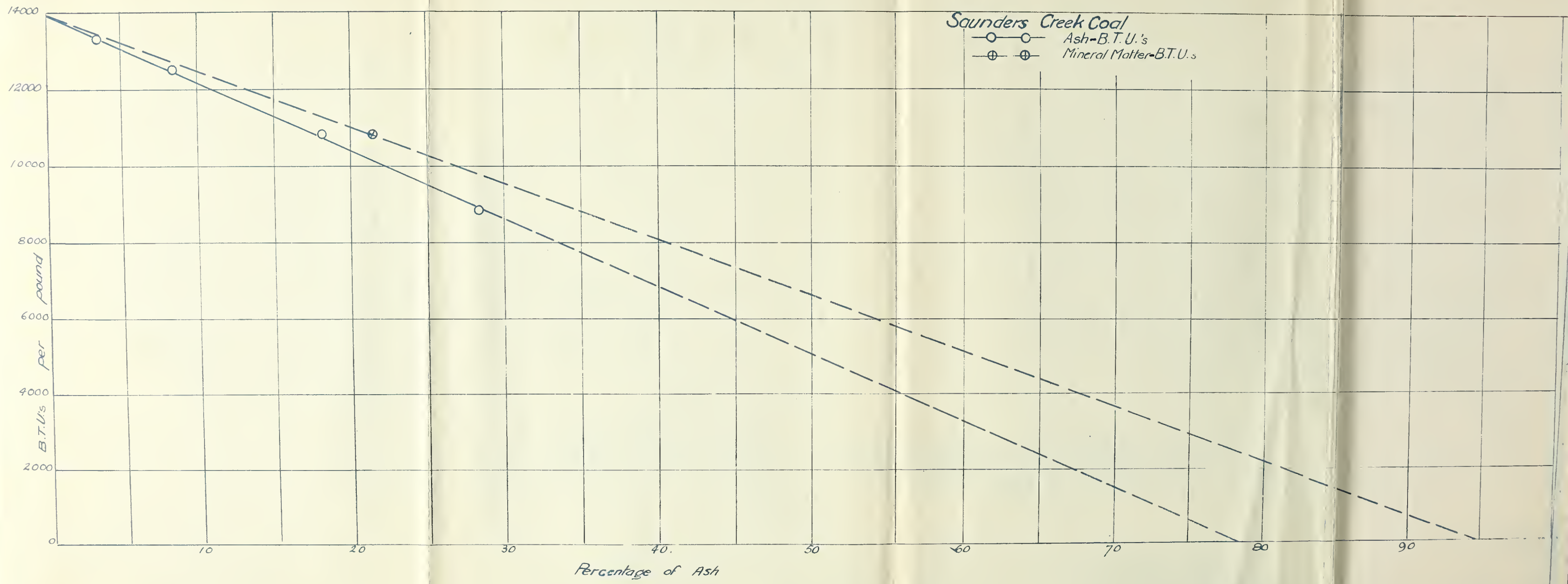
information regarding the distribution of the moisture in the separated portions, since, in common with low moisture coals, the moisture is well retained. This information was desired in connection with a problem in Chapter I. The former, the high proportion of inert volatile matter, gave promise that the compounds causing this loss would be in such quantity that they could be readily detected.

Heavy Solution Curve

The heavy solution curve was first determined for the coal, (Fig. 11, page 71). One of the points determining the curve was obtained by analysis of the large sample of sink referred to above. Determination of carbon dioxide present as carbonates, sulphur in pyrites, and sulphates were next made on the large sample of sink.

Quantitative Analyses

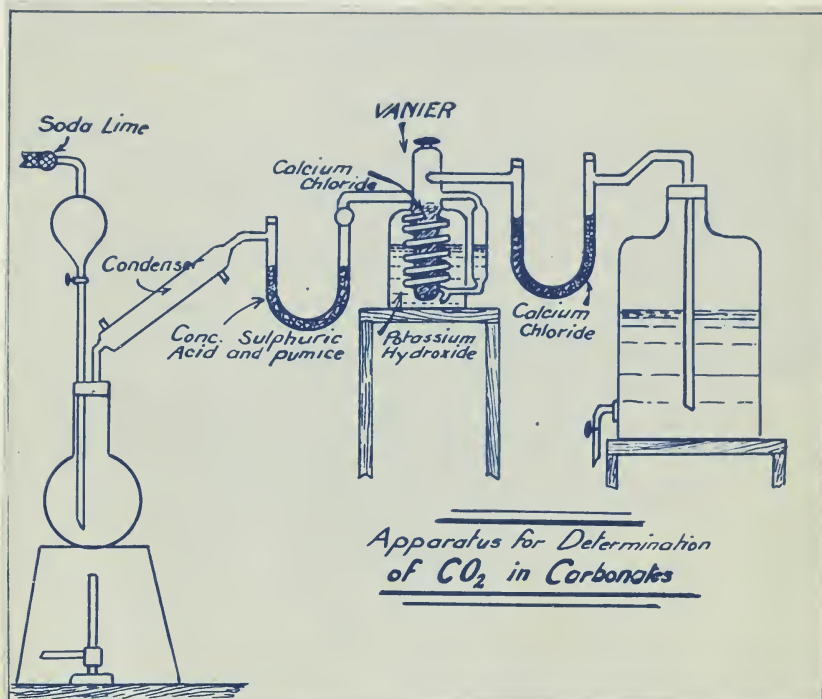
Carbon Dioxide from Carbonates:- Carbon Dioxide from carbonates was determined by treating the powdered coal with sulphuric acid and collecting the gas liberated in potassium hydroxide. The apparatus was set



up as shown in Figure 10, page 73.

About 10 grams of coal was placed in the carbon dioxide flask. The dropping funnel was filled with 60 c.c. of a 3% solution of sulphuric acid. A rubber stopper carrying a calcium chloride tube filled with soda lime prevents carbon dioxide from the air entering the flask. A wide delivery tube with condenser attached carries off the gases liberated when the acid is made to drop on the coal. Any vapour which might distil over is absorbed in the "vanier" bulb, containing potassium hydroxide. A guard tube filled with calcium chloride prevents moisture from diffusing back from the aspirator bottle. The determination is made by dropping the acid slowly on the coal; when all the acid has been added the end of the dropping funnel should be sealed off by the acid. The gases liberated are aspirated through the absorption train. The water to the condenser is turned on and the flask slowly heated to the boiling point and kept gently boiling for half an hour. At the end of that time the heating is stopped and 250 c.c. more of air is aspirated through the system. The vanier is then disconnected and after remaining half an hour beside

Fig. 10.



the balance is weighed, the increases in weight being equal to the carbon dioxide liberated from the coal. Carbon dioxide in the ash is determined in the same way.

Pyritic Sulphur:- Pyritic sulphur is determined by treating 1 gram of the powdered coal, at room temperature, for four days, with 40 c.c. of nitric acid, 1.12 specific gravity. At the end of the treatment the solution is filtered and the sulphate is precipitated with a 10% solution of barium chloride. From the sulphur obtained, less the sulphur present as sulphates, determined as below, the amount of pyrites is calculated. (1).

Sulphur from Sulphates:- For sulphate sulphur, the method is as follows:

5 grams of finely powdered coal are digested for 40 hours at 60° C. in 300 c.c. of a 3% solution of hydrochloric acid. After the treatment the solution is filtered, and the sulphur is precipitated in the usual manner, by barium chloride solution. The sulphur present in the coal is calculated to sulphur trioxide. One outstanding feature of Alberta coals that should be mentioned is the low sulphur content.

(1) A.R.Powell, "The Analysis of sulphur forms in coal" Tech.Paper #254, Bureau of Mines, U.S.A. p.8-1921

Extraction

As a check on the above determinations for mineral matter, another part of the same sample, 6 grams approximately, was treated as follows:- the coal was placed in a beaker, covered with a thin film of para^{or} wax. 30 c.c. of normal hydrochloric acid were added. The coal was set aside for 6 days. At the end of the time the clear liquid was decanted off and the coal washed by decantation, care being taken to account for coal lost in decantation, by pouring the liquid off through a filter and weighing the coal lost when dry. To the wet coal was then added 5 c.c. of C. P. hydrofluoric acid and it was again set aside for 6 days and at the end of the period was thoroughly washed by decantation. The coal was then placed in the air-drying apparatus for twenty-four hours, in order to bring it as nearly as possible to its former state of moisture content. Analyses were then made on the coal for moisture, ash and calorific value. The loss in weight plus the ash was then checked up with the mineral content determined above.

R e s u l t s

The accompanying table (XVI, next page) gives the results of the analyses.

Qualitative Tests:- Qualitative tests carried out on the hydrochloric and hydrofluoric acid extracts show the presence of calcium, with some iron and a trace of magnesium. From the high carbonate value for the coal it would appear that calcium carbonate was the chief mineral constituent in the coal.

Ash to Mineral Matter:- Having determined by quantitative analyses the known inert volatile constituents in the special sample of sink mentioned above, their value can now be added to the value of the ash of this same sample, and the new value (if all the inert volatile constituents have been accounted for) should equal the mineral matter in the sample. If this new value be plotted against the original calorific value, on the same graph as the heavy solution curve, a line drawn through the point for "pure coal" and the point for the mineral matter value of the sample, should cut the ash axis, or rather in this case, the mineral matter axis, at 100% for zero calorific value. For Saunder's Creek coal, it is seen that the mineral

TABLE XVI.- ANALYSIS OF SAUNDERS CREEK COAL

(A) Proximate Analysis for Heavy Solution Curve

Type of Sample	Air-Dried Basis			Dry Basis	
	Moisture, %	Ash, %	B.T.U./lb.	Ash, %	B.T.U./lb.
Original	7.26	7.71	11,613	8.32	12,522
Float	5.58	3.11	12,590	3.29	13,334
Sink	3.23	27.36	8,530	28.28	8.815
Special Sink	5.30	17.14	10,236	18.11	10,809

Calorific Value of Pure Coal, B.T.U. per lb. = 13,930

Zero calorific value at 78.5% Ash, ∴ Mineral Matter Factor = 1.27,

Ash corrected to Mineral Matter = $18.11 \times 1.27 = 23.08\%$

(B) Quantitative Determination of Inert Constituents

Ash in dry sample %	18.11	
Less 2/3 pyrites %	0.00	18.11
CO ₂ from carbonates %	4.00	
Less CO ₂ in the ash %	0.75	3.25
Pyrite %		0.00
Sulphur Trioxide from sulphates . . %		0.00
Total Inert Matter %		21.36

(C) Extraction of Mineral Matter with HCl and HF

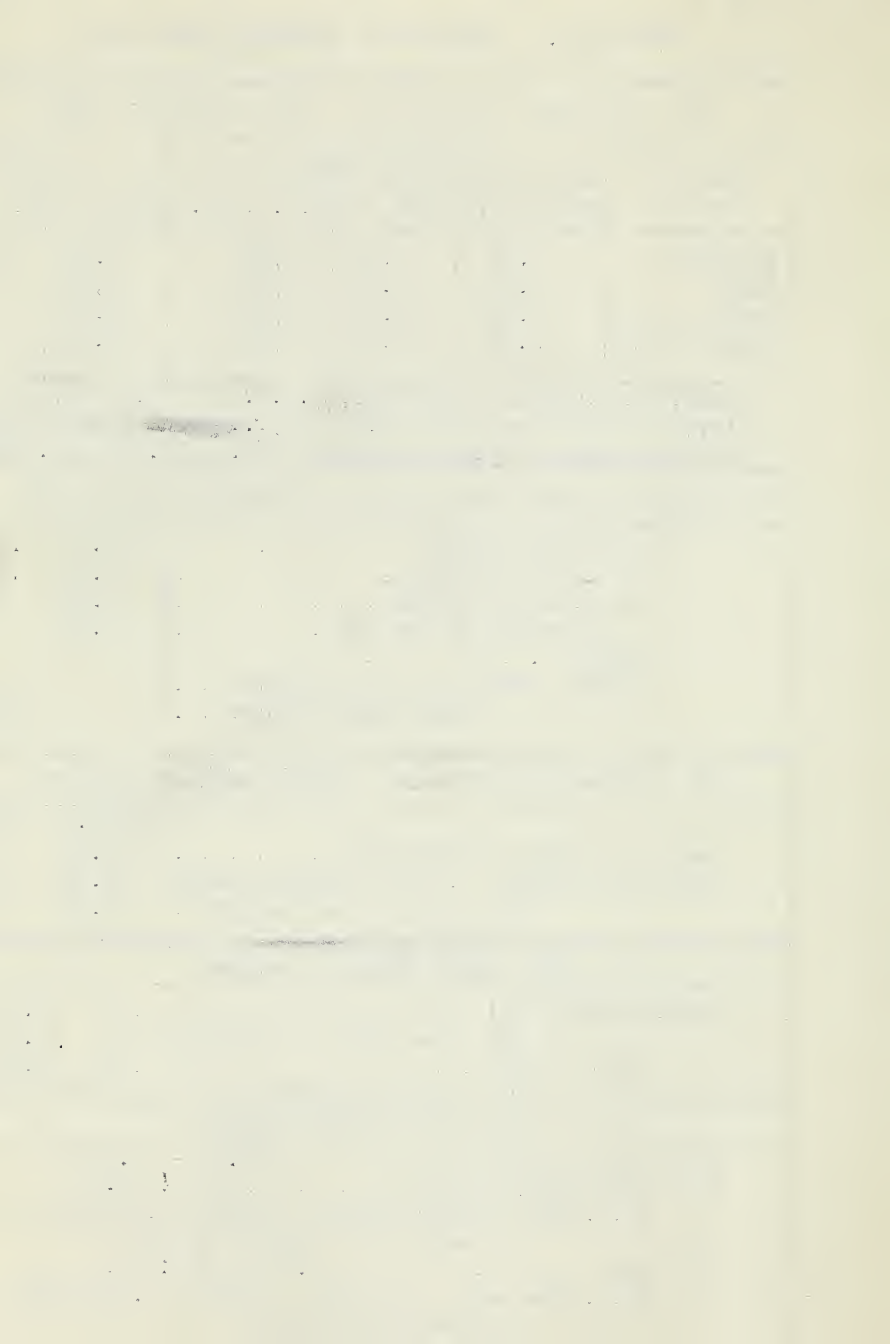
	Expt. 1	Expt. 2
Loss in weight on extraction %	18.22	18.03
Ash from residue . . % of original weight	2.91	3.38
Total Inert Constituents . . . %	21.13	21.41

(D) Total Mineral Matter

Determined by (A) %	23.08
" " (B) (less water of hydration) %	21.36
" " (C) %	21.27

(E) Water of Hydration

% of Coal, dry basis	$23.08 - 21.36 =$	1.72
% of Total Mineral Matter	$1.72 \times 100 \div 23.08 =$	7.45
(i.e. the coal, dry basis, contains 0.08% water of hydration in each 1% of mineral matter)		
% of Ash	$1.72 \times 100 \div 18.11 =$	9.49
(i.e. the coal, dry basis, contains 0.10% water of hydration for each 1% of ash)		



matter value, as determined, does not account for all inert matter, as the line cuts at 92.6%. The only volatile inert constituent which has not been accounted for is water of hydration, so that the difference between 100 and 92.6% might be the measure of the water of hydration present in this coal.

Ultimate Analysis:- A further test was carried out as follows:

From the samples used in the determination of the heavy solution curves, two were selected, one of very low ash content and one of high, and on these determinations were made for carbon and hydrogen content. It was thought that since the impurities in the coal were high in carbonates, that the value of the carbon-hydrogen ratio of the two samples should differ; the samples with the higher ash giving the higher ratio, since the carbon dioxide given off from the carbonates will be added to the carbon of the coal substance. The results obtained point that way.

<u>Dry Basis</u>	<u>Carbon</u>	<u>Hydrogen</u>	<u>Ash</u>	<u>C/H.</u>
Float	76.60	4.01	3.34	19.11
Sink	50.80	2.43	31.23	20.90

From these results it can be seen that the mineral or inorganic constituents of a coal have a very important

bearing on the ultimate analysis.

Nordegg Coal.

The next coal treated was a coal from Nordegg, a bituminous coal, from the Kootenay horizon. The heavy solution curve was first obtained (Figure 12, next page) and all the analyses made on the Saunder's Creek coal were carried out on this coal. Slight changes only were made in some of the methods of analyses because of the different nature of the coal, but no essential changes were made. Analyses for carbon dioxide, pyritic sulphur and sulphate sulphur were made as before. The sample chosen for special analyses contained 28.3% ash. The reduction in ash brought about by hydrochloric and hydrofluoric acid extraction was very slight compared with the reduction in the Saunder's Creek coal.

Results. The results of the analyses are shown on Table XVII. page 81.

Qualitative Tests:- Qualitative tests on the hydrochloric and hydrofluoric extracts showed the presence of iron, aluminium and a trace of calcium.

If, as in the case of the Saunder's Creek coal, the ash is corrected to mineral matter and the value plotted against the calorific value, the straight line

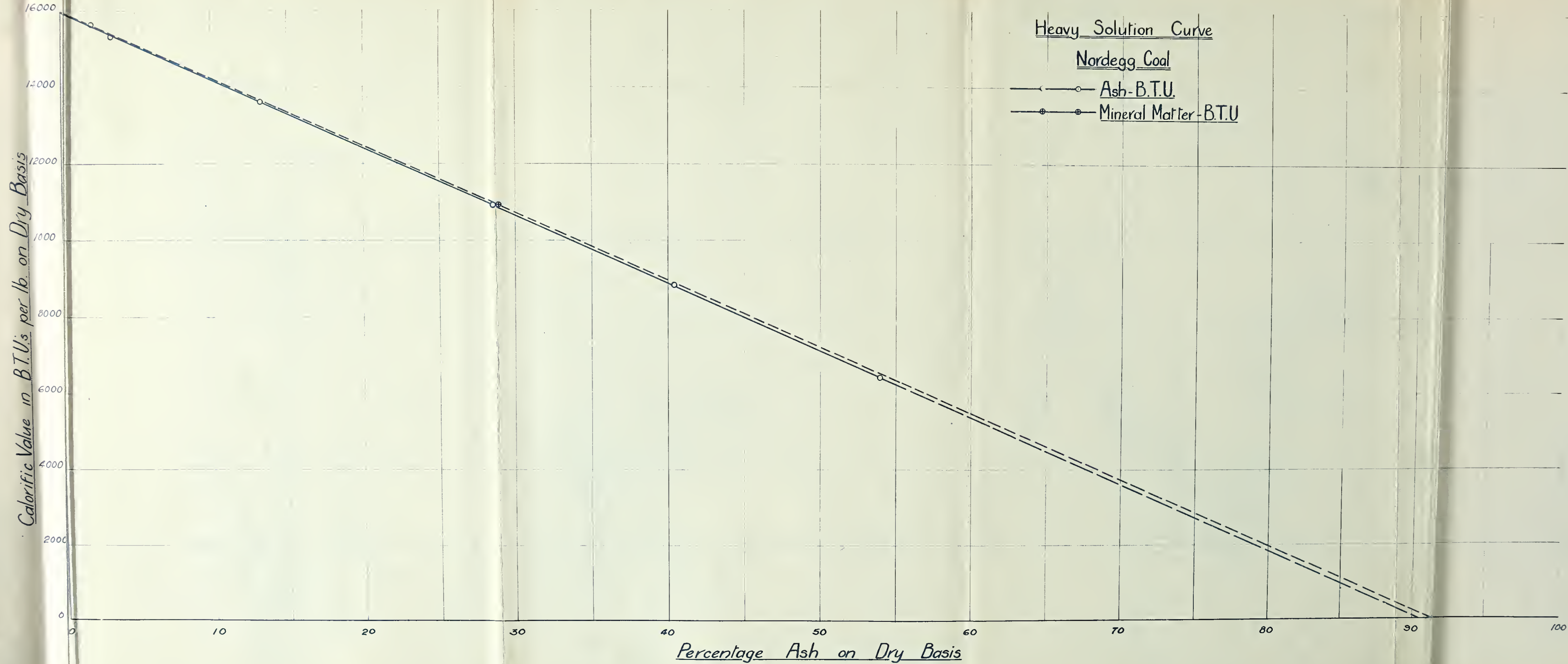


TABLE XVII.- ANALYSIS OF NORDEGG COAL, KOOTENAY HORIZON

(A) Proximate Analysis for Heavy Solution Curve					
Type of Sample	Air-Dried Basis			Dry Basis	
	Moisture, %	Ash, %	B.T.U./lb.	Ash, %	B.T.U./lb.
Float	0.45	2.09	15,544	2.10	15,610
Float	0.47	3.38	15,237	3.40	15,309
Original	0.41	13.22	13,551	13.28	13,607
Special Sink	0.71	28.31	10,848	28.50	10,926
Sink	0.63	40.21	8,782	40.42	8.838
Calorific Value of Pure Coal, B.T.U./lb. = 15,950					
Zero calorific value at 90.3% Ash, ∴ Mineral Matter Factor = 1.108					
Mineral Matter in Special Sink = 28.50 x 1.11 = 31.62					
(B) Quantitative Determination of Inert Constituents					
Ash in dry sample %			28.50		
Less 2/3 pyrites %			<u>0.56</u>	27.94	
CO ₂ from carbonates, etc. %				0.15	
Pyrites (from pyritic sulphur) . . . %				0.84	
Sulphur Trioxide from sulphates . . %				<u>0.01</u>	
Total Inert Matter, less water of hydration				28.94%	
(C) Extraction of Mineral Matter with HCl and HF					
			<u>Expt. 1</u>	<u>Expt. 2</u>	
Loss in weight on extraction %			7.15	7.34	
Ash from residue . . % of original weight			<u>23.08</u>	<u>23.29</u>	
Total Inert Constituents %			30.23	30.63	
(D) Total Mineral Matter					
Determined by (A) %			31.62		
" " (B) (less water of hydration) . %			28.94		
" " (C) %			30.43		
(E) Water of Hydration as					
% of Coal, dry basis 31.62 - 28.92 = 2.70					
% of Total Mineral Matter 2.65 x 100 ÷ 31.62 = 8.38					
(i.e., the coal, dry basis, contains 0.08% water of hydration in 1% mineral matter)					
% of Ash 2.65 x 100 ÷ 28.50 = 9.30					
(i.e., the coal, dry basis, contains 0.09% water of hydration for each 1% ash)					

joining the pure coal value and this point, should, on being produced, cut the horizontal axis at 100%, if all the inert matter has been accounted for. It should be noticed that the unaccounted for loss amounts to about 10%.

Ultimate Analysis:- Again as in the Saunder's Creek coal, two samples used in the determination of the heavy solution curve were selected, one having low ash content and the other high. On these samples determinations for carbon and hydrogen were made. While the carbon dioxide present as carbonates is small the unaccounted for loss is quite large, almost 10%. If this loss is water of hydration, the carbon-hydrogen ratio will be low for the coal of high ash content. The water of hydration liberated in the determination of the carbon and hydrogen has not been included in the amount of water determined at 105° C. It will therefore be accounted for as water formed from hydrogen in the coal, which will give too high a value for the hydrogen. This conclusion the analyses support. The greater the ash content the greater will be the error.

<u>Dry Basis</u>	<u>Carbon</u>	<u>Hydrogen</u>	<u>Ash</u>	<u>C/H</u>
Float	87.73	4.12	2.07	21.79
Sink	51.20	2.80	40.46	18.30

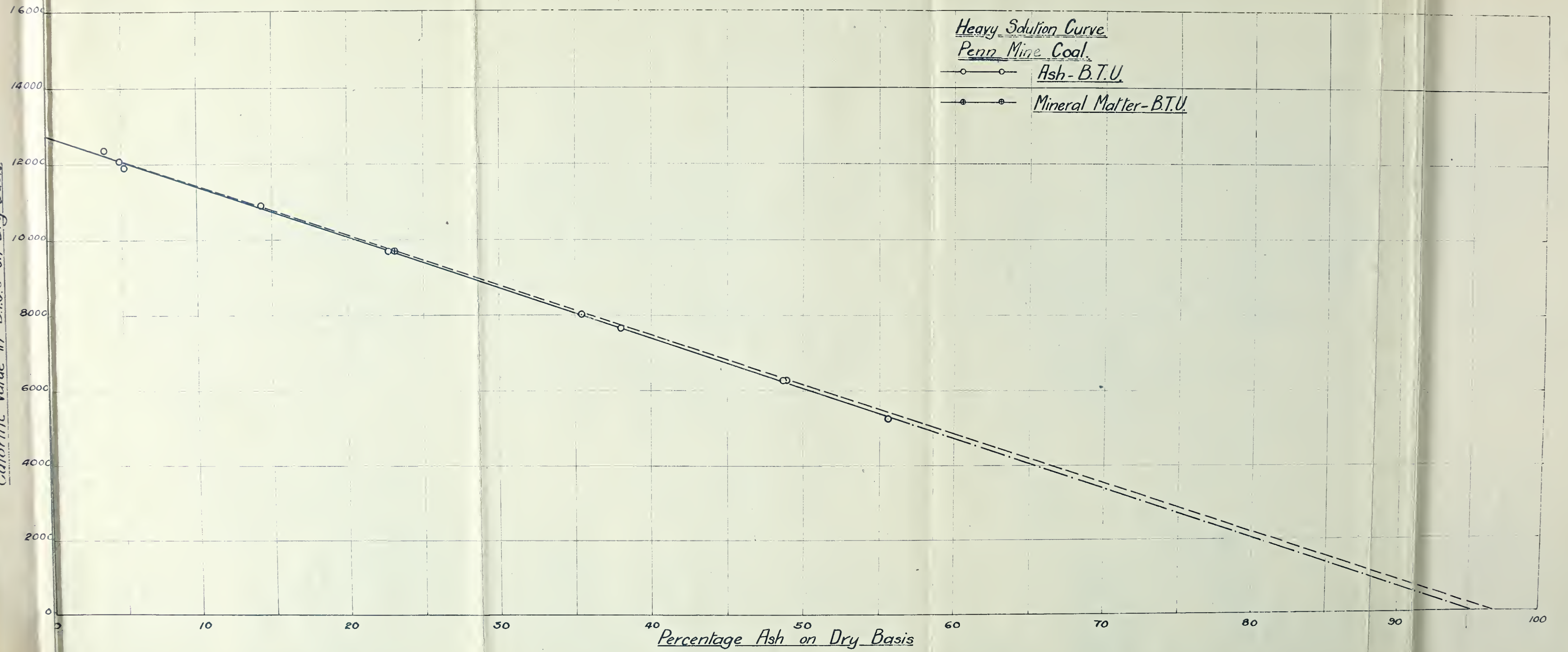
Edmonton Coal.

The third coal treated was an Edmonton coal, from Penn Mine, Edmonton. The heavy solution curve was first obtained by the usual method (Figure 13, next page), and the other analyses were made on a sample having 22.72% ash on a dry basis. The accompanying Table (XVIII, page 85) shows the results obtained.

Qualitative Analysis:- Qualitative analysis of the acid extract shows the presence of some iron, aluminium and a trace of calcium.

No ultimate analysis was made for carbon and hydrogen on the Edmonton coal.

Calorific Value in B.T.U.'s on Dry Basis



Heavy Solution Curve

Penn Mine Coal.

—○—○— Ash - B.T.U.

—●—●— Mineral Matter - B.T.U.

Percentage Ash on Dry Basis

TABLE XVIII.- ANALYSIS OF PENN COAL

(A) Proximate Analysis for Heavy Solution Curve

Type of Sample	Air-Dried Basis			Dry Basis	
	Moisture, %	Ash, %	B.T.U./lb.	Ash, %	B.T.U./lb.
Float	10.92	3.46	11,026	3.88	12,377
Float	8.74	4.46	11,019	4.89	12,074
Float	10.21	4.67	10,680	5.20	11,900
Original	12.10	12.58	9,599	14.31	10,920
Special Sink	9.55	20.55	8,784	22.72	9,712
Sink	7.14	31.86	7,450	35.39	8,023
Sink	5.79	45.84	5,918	48.65	6,282
Sink	5.08	52.80	4,981	55.62	5,248
Sink	7.38	45.27	5,823	48.90	6,287
Sink	7.65	35.22	7,076	38.13	7,662

Calorific Value of Pure Coal, B.T.U./lb. = 12,740

Zero calorific value at 95.5% Ash, ∴ Mineral Matter Factor = 1.047

Mineral Matter in Special Sink = $22.72 \times 1.047 = 23.81\%$

(B) Quantitative Determination of Inert Constituents

Ash in dry sample	%	22.72	
Less 2/3 pyrites	%	<u>0.00</u>	22.72
CO ₂ from carbonates less CO ₂ in ash . . .	%		0.39
Sulphur Trioxide, from sulphates . . .	%		<u>0.00</u>
Total Inert Constituents . . .	%		23.11

(C) Extraction of Mineral Matter with HCl and HF

	Expt. 1	Expt. 2
Loss in weight on extraction	9.13	12.19
Ash from residue. . % of original weight	<u>8.84</u>	<u>11.97</u>
Total Inert Constituents . . . %	21.32	20.81

(D) Total Mineral Matter

Determined by (A)	%	23.81
" " (B) (less water of hydration) . .	%	23.11
" " (C)	%	21.07

(E) Water of Hydration as

% of Coal, dry basis	$23.81 - 23.11 = 0.70$
% of Total Mineral Matter . . .	$0.70 \times 100 \div 23.81 = 2.94$
(i.e., the coal, dry basis, contains 0.03% water of hydration in 1% mineral matter)	
% of Ash	$0.70 \times 100 \div 22.72 = 3.08$
(i.e., the coal, dry basis, contains 0.03% water of hydration in 1% of ash)	

C o n c l u s i o n s

In the work which has just been described, three methods have been employed. Only on the first two can any reliance be put as a means of determining how much volatile mineral matter has been lost in the determination of ash in coal, or what the quantity of mineral matter in a coal is.

Extraction by hydrochloric and hydrofluoric acid can only be of value if the mineral matter still remaining in the coal will not give up volatile inert matter, in determining the ash. Proof is lacking on this point. The extraction in all three coals was accompanied by a lowering of the calorific value of the coal 300 to 400 B. T. U's per lb. This suggests, of course, removal of coal substance along with the mineral matter. In the case of the Saunder's Creek coal the results of duplicate determinations agree remarkably well with each other, as well as agreeing (see Table XVI) with the result obtained by direct determination, but the results of the other coals, Nordegg and Edmonton, show values higher and lower, respectively, than the results obtained by direct determinations. The method, therefore, cannot

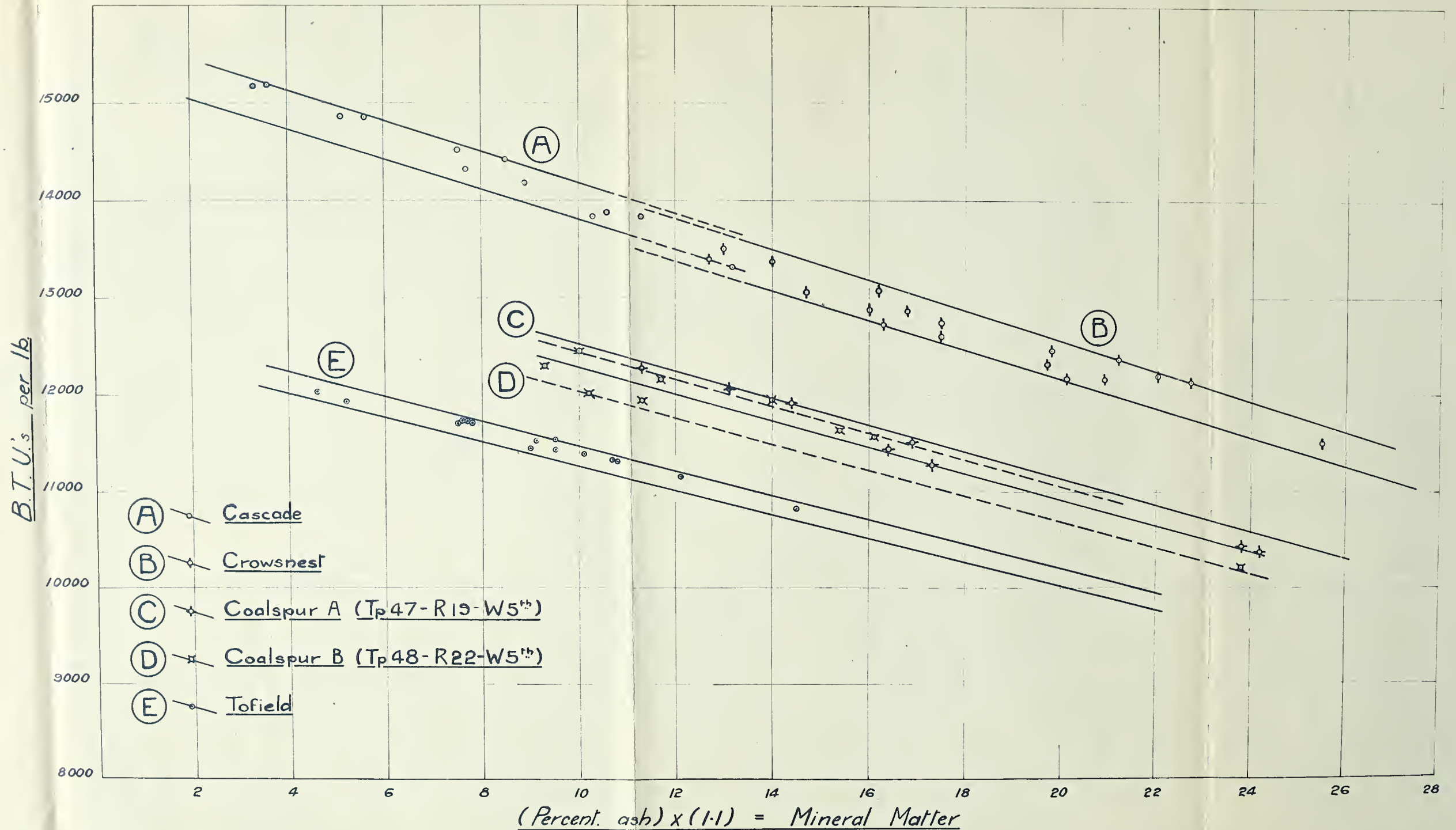
be regarded as accurate, but might be used as a rough check on the other work.

Of the methods which have significance as a means of determining the amount of mineral matter loss on combustion of a coal, the first gives no information as to the nature of the compounds; it merely indicates the total amount which has been lost in the process of determining the ash. In the three coals treated, different values were obtained for this loss, namely, 21.5%, 9.7% and 4.5%. Different values have been obtained for other coals, but quite a number of Alberta coals appear to suffer a loss of around 10%. Parr and Wheeler, elsewhere mentioned in their work on "Unit Coal, use a constant factor of 1.08 in correcting ash as determined to mineral matter. This corresponds to a loss of about 7.4% of volatile inert matter. But if the carbonates and chlorides together are found to be more than 0.5%, these must be determined separately and the correction made as before for other inert volatile matter lost, besides carbon dioxide. As assumption is made by Parr in using this factor that "pure" shale has a constant volatile content of 7.4%. (¹). Drakeley, in the article just referred to, gave (¹) Jour. Soc. Ind. Chem. p. 136 T. May 2, 1924.

results of some tests on what was considered pure shale and placed the value at 9.5%. In later determinations, however, by more complete drying, he obtained the value of 7.2%.

"Crude coal," it has been said, "may be regarded as a mixture of coal and shale." (¹). The results obtained by the heavy solution curve for any one coal support this view, as the fractions of coal, separated by heavy solutions, from a single sample, behave as though they consisted merely of different proportions of the two same constituents, namely "pure coal" and "pure mineral matter". Even different samples from the same area appear to be made up of a uniformly pure coal substance and a uniformly pure shale, in that when the values of their calorific value and ash are plotted in the same way as for calorific value-ash heavy solution curves, they tend to lie in a straight line. In the accompanying Figure 14, next page, four different types of coal are represented; one type is divided into two on the basis of the townships from which the samples came. The ash is corrected to mineral matter by multiplying by 1.1

(¹) Jour. Soc. Ind. Chem. p.278 T. Aug 22, 1924.



The pairs of lines shown are drawn so that they pass through the point for 100% mineral matter and zero calorific value, and just include between them all the points of the group. When ash is plotted instead of mineral matter, the pairs of lines are further apart. Correcting for mineral matter has the effect of bringing the lines closer together, i.e., the points conform more nearly to the linear relation found by analysis of fractions of a single sample from the area, separated by heavy solution treatment.

In view then of what has been said above regarding the volatile matter content of shales, it follows that there cannot be such a thing as 100% ash. If such a thing as a coal, could be imagined, in which the pure coal content was so small that the shale was practically 100%, the ash from this "coal" would still be less than 100%, for according to the values quoted for the volatile matter in shale, the ash would range from 90.5 to 92.8%. For this reason the incorrectness of the term "corrected ash" already referred to becomes evident.

From the results obtained in the tests carried out, this fact is plain, that both pure coal and mineral matter may vary for different coals. The

calorific value of the pure coal as determined by calorific-ash heavy solution curves, differs in all three coals tested, also the nature and amount of volatile inert constituents in the mineral matter. As a means, therefore, of giving a measure of the total inert volatile constituents of the mineral matter in coal and furnishing a factor by which ash may be corrected to mineral matter, the heavy solution method is quite satisfactory, but as was pointed out previously, it does not tell anything regarding the nature of the constituents which make up the inert volatile matter.

It has been found that for a number of Alberta coals the loss, as determined by calorific-ash heavy solution curves, is in the neighborhood of 10%. This means that for these coals, a factor of 1.1 should be used in correcting ash to mineral matter. The Saunder's Creek coal was chosen in beginning the tests for its high volatile mineral matter loss, but it is a coincidence that the other coals tested gave different values and that the highest and lowest values were obtained for coals tested in the S. & I.R.C.A. laboratory. The highest value is for Saunder's Creek, the lowest for the Edmonton coal, their factors being

1.27 and 1.05 respectively. The third coal, from Nordegg, has a factor of 1.1 which is common to a number of Alberta coals.

By the second method the amount and type of inert constituents can be determined, except water of hydration. The latter constituent will be dealt with shortly. The determination of the volatile mineral matter is of much importance in connection with the ultimate analysis of coal. In coals where the mineral impurities are high, largely carbonates or clays and shales, determination should be made for volatile mineral matter. Only carbon dioxide, however, can be determined by direct analysis; water of hydration from clay and shale in coal cannot be. The presence of either of these constituents in any quantity in coal will make an appreciable difference in the values obtained for the carbon and hydrogen content of the coal. The ultimate analysis made above on Saunder's Creek and Nordegg coal bring out the discrepancies in the carbon-hydrogen ratio.

An indirect method for the determination of water of hydration is suggested by a combination of methods 1 and 2 (outlined above) This will be dealt with in the next section under "Water of Hydration".

W A T E R O F H Y D R A T I O N

The close relation of "Water of Hydration" to mineral matter is the reason for its discussion being deferred until now. Though truly water, it is not determined by any of the means for determining water in coal outlined in Chapter I. of this thesis.

H i s t o r i c a l

In 1909 S.W. Parr (¹), already mentioned, in calculating the calorific value of "unit coal" uses a factor of 1.08 in correcting ash to mineral matter; this factor is supposed to include water of hydration as well as other volatile mineral constituents.

Illingworth (²) has said of water of hydration, "No reliable method exists for its determination," so that in calculating ash to a mineral basis he takes no account of this constituent.

E. Stansfield (³) has used the factor 1.10

(¹) S.W.Parr & W.F. Wheeler-"Unit Coal",University of Illinois Bulletin #37,p.12-1909.

(²) S.Roy Illingworth-"Analysis of Coal and its By-Products", p.74, 1921.

(³) E. Stansfield, Bulletin C.I. M & M. Vol. 156, 1925.

in correcting the ash of Alberta coal to mineral matter. This factor also includes water of hydration.

Water of Hydration from Heavy Solution Curve:

It was pointed out elsewhere that the analyses for volatile mineral matter in the coals tested were carried out on a large sample of sink, one for each coal, the ash and calorific values of each sink determining a point on their respective heavy solution curves. It was thought that if to the value of the ash, as determined, of this sample, were added the values of the determined volatile mineral matter lost, the amount of water of hydration could be estimated by joining this point to the pure coal point, and producing the line to meet the ash axis at the point for zero calorific value. The difference between the point of intersection and 100% ash being the unaccounted loss, is presumably the water of hydration in 100% of mineral matter. The result could also be obtained by calculation, knowing the value of the ash for zero calorific value, the percentage of ash and the percentage of ash corrected to mineral matter (less water of hydration).

e. g.,

Saunder's Creek Coal.

Ash for zero calorific value 78.5%

Ash as determined 18.11

Mineral matter less water of hydration 21.36

$$18.11 : 21.36 = 78.5 : X$$

$$X = \frac{21.36 \times 78.5}{18.11}$$

$$= 92.6$$

$$\text{Water of Hydration?} = 7.4$$

The following values were obtained:

	Loss not accounted for	
	100% M.Matter	1% M.Matter
Saunder's	7.45	= 0.075
Nordegg	8.54	= 0.085
Edmonton	2.94	= 0.029
<u>Water of hydration from ultimate analysis.</u>		

The question has been raised already, is this unaccounted loss water of hydration, or not? It was pointed out earlier in this thesis that differences in carbon-hydrogen ratio might be explained by differences in the amounts of water of hydration or carbonates present in the samples. In looking for a proof to support the assumption that the unaccounted loss as shown by the mineral matter-calorific value curve,

was water of hydration, it was suggested that the ultimate analysis made on the Nordegg and Saunder's Creek coal might throw some light on the subject. Each pair of samples analysed, being the same coal, ought to have the same carbon-hydrogen ratio, for the pure coal substance. If we assume that the difference in the values of the carbon-hydrogen ratio are caused by the mineral matter in the coal, then by making corrections in the carbon and hydrogen as determined, for differences due to mineral matter, the ratios should be equal, which means that the values of the results obtained by analysis may be equated. The corrected ratios will then become equal to:-

the total carbon in the coal, less the carbon from the mineral matter, divided by the total hydrogen in the coal less the hydrogen due to mineral matter.

The carbon due to mineral matter can be determined, and is equal to the percentage of ash multiplied by the percentage of carbon dioxide associated with 1% ash, multiplied by the ratio of carbon to carbon dioxide. The hydrogen due to mineral matter cannot be determined, but is equal to the percentage of ash multiplied by the ratio of

two atoms of hydrogen to water, multiplied by the percentage of water of hydration for 1% ash.

Nordeg Coal.

Dealing with the Nordeg coal:

<u>Sample</u>	<u>Carbon</u>	<u>Hydrogen</u>	<u>Ash.</u>
A.	87.73	4.12	2.10
B.	51.20	2.80	40.46

Carbon dioxide from carbonates = 0.005 for 1% ash.

Let X = the water of hydration for 1% ash.

(The correction for carbon dioxide from carbonates was neglected)

The equation is as follows:

$$\begin{array}{ccc} \text{A.} & & \text{B.} \\ \frac{87.73}{4.12} - (2.10 \times 1/9 X) & = & \frac{51.20}{2.80} - (40.46 \times 1/9 X) \end{array}$$

On simplifying the equation,

$$X = 0.048.$$

Water of Hydration for 1% ash.

By ultimate analysis 0.05%

By calorific ash curve 0.09%

Saunder's Creek Coal.

<u>Sample</u>	<u>Carbon</u>	<u>Hydrogen</u>	<u>Ash.</u>
A.	76.60	4.01	3.34
B.	50.80	2.08	31.23

Carbon dioxide from carbonates = 0.31% for 1% ash.

Let X = water of hydration for 1% ash.

The equation is as follows:

$$\begin{array}{cc} \text{A.} & \text{B.} \\ 76.60 - \left(\frac{400 \times 3.34}{18.11} \times \frac{12}{44} \right) & 50.80 - \left(\frac{400 \times 31.23}{18.11} \times \frac{12}{24} \right) \\ \hline 4.01 - (3.34 \times 1/9 X) & 2.08 - (31.23 \times 1/9 X) \end{array} =$$

On simplifying

$$X = 0.15.$$

Water of Hydration for 1% ash.

By ultimate analysis - 0.15

By calorific ash curve + 0.10

The results are not all that could be desired. The value obtained for the Nordegg coal approximates the value obtained by the calorific value-ash curve, but of the results for the Saunder's Creek coal, not much can be said. It should be pointed out, however, that not all the determinations made on the samples for carbon and hydrogen were run in duplicate. A small error in the hydrogen determination would make a large error in the final result. Sufficient work has not been done on the method to pass final judgment. This work, however, will be continued.

B I B L I O G R A P H Y.

1. American Soc. Test. Mat.- Standards 1924.
2. A. S. T. M. Standards-Vol.XIV. pt.1, Committee Reports - 1914.
3. Chemistry & Industry-Vol. 43.No.18.pp 135 T & 136 T. May 2, 1924.
4. Ellworthy, R.T. "Natural Gas in Canada and its Uses"-C. I. M. & M.Bulletin # 155-Mar. 1925.
5. Hoffman,G.- "Hygroscopicity of Some Canadian Fossil Fuels"-Trans.R.Soc. Can. 1st.Series, Vol.VII.Sec.3- 1889.
6. Holloway & Coste-Report of 10th Sub-Committee to 8th Inter. Congress on Applied Chemistry.
7. Hillebrand Dr.-Jour. Am.Chem.Soc. Vol.20,p 281-1898.
8. Illingworth, S.Roy- "The Analysis of Coal and its By-Products", pp 19, 84.
9. Jour. Ind.Eng.Chem.-Vol 5, June 1913.
10. Jour. Am.Chem.Soc.-Vol 21-1899.
11. Jour. Am.Chem.Soc.-Vol.27,p.631-1906.
12. Jour.Soc.Chem & Ind.-Vol.XIX.#2,p 61, Jan.31,1911.
13. Jour. Ind.Eng.Chem. Vol.I. p 65, Feb. 1909.
14. Jour. Soc. Ind.Chem. p 278 T.-Aug. 22, 1924.

15. Lewis, J.W.-"A further study of Alberta Coal"-
Thesis for M.Sc. degree U. of A.
16. Porter, J.B. & others-"Investigation of the Coals
of Canada"-Vol.II. Part IX.
17. Parr, S.W. & Wheeler, W.F.-"Unit Coal and the
Composition of Coal Ash", University of Illinois,
Bulletin # 37, p 5- 1909.
18. Powell, A.R.-"The Analysis of Sulphur forms in
Coal"-Bureau of Mines,Tech.Paper # 254,U.S.A.
p. 8, 1921.
19. Stopes, Marie & Wheeler,W.F. -"Fuel in Science
and Practice-Vol. II. # 2,p 37-1923.
20. S. & I.R.C.A.-Third Annual Report, p 39-1922.
21. Stansfield E.,C.I. M. & M., Bulletin #156-
April 1925.
22. Stansfield E., Summary Report, Mines Branch,
Dept. Mines Ottawa, p 38, 1919.
23. Stansfield E.,Summary Report, Mines Branch,
Dept. Mines, Ottawa, p 123- 1911.
24. Stansfield E. & Nicolls,G.H.H.-"Analysis of
Canadian Fuels"- part 4, 1922.
25. Stanton,F.M. & Fieldner,A.C.-"Methods of
Analysing Coal & Coke", Tech.Paper #8,
Bureau of Mines,U.S.A.-1912

26. S. & I.R.C.A.-Fourth Annual Report-1923.



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